

JPL Contract 951003
NASA Contract NAS7-100

STUDY OF THE EFFECTS OF ETHYLENE OXIDE-FREON 12 UPON PROPERTIES OF POLYMERS AND METALLIC SURFACES

FINAL REPORT COVERING THE PERIOD
10 October 1964 through 31 March 1966

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Pages 7 & 10 Revised 1 June 1966

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Contract NAS7-100.

FOREWORD

This report summarizes work performed under Jet Propulsion Laboratory Contract 951003, NAS 7-100, "Study of Effects of Ethylene Oxide - Freon 12 upon Properties of Polymers and Metallic Surfaces" during the period October 1, 1964 through March 31, 1966. Mr. H. Harvey was JPL Cognizant Engineer during the first year of the program, and Mr. D. Kohorst for the remainder.

The Hughes Project Engineer was R. F. Rydelek who was also responsible for the Materials Screening portion of the program. The Mechanisms Studies were conducted by Dr. A. L. Landis. D. Fraser aided in the performance of both the Mechanisms and Screening portions of the program.

Acknowledgment is given to the many members of the Materials Technology Department who aided in the conduct of the program, especially to Dr. S. Wiener and Dr. D. C. Smith for guidance and technical support.

ABSTRACT

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Sixty-eight commercial polymeric products were exposed to the following two-step spacecraft sterilization procedure: 48 hours in an atmosphere of 12 percent ethylene oxide - Freon 12 sterilant gas at 30-50 percent relative humidity followed by 108 to 120 hours at 145°C under a dry nitrogen atmosphere. Physical, mechanical, and electrical tests were performed on materials to determine the amount of degradation caused by each step and to establish the compatibility of products with the entire procedure. A concurrent mechanism study was conducted to provide insight into the nature of the gas-material interaction.

Although all products showed a change in properties after gas exposure, and an additional change after dry heat cycling, only two were rated "not compatible", and 18 as "marginal" by the criteria established by JPL. None of the products tested showed an appreciable loss of electrical properties.

The mechanism study indicated that both pure and commercial products are capable of adsorbing up to four percent by weight of sterilant gas. Compounds released all but a fraction of a percent of the adsorbed gas when exposed to a vacuum. In most cases, exposure to dry heat cycling after gas and vacuum exposure produced a net weight loss in test materials. The main gas-material interaction that occurs during gas exposure is a reversible physical adsorption.

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I. INTRODUCTION

A. OBJECTIVES

The advent of planetary probes and landing vehicles has focused attention on the need for biological sterilization of spacecraft. One of the major mission objectives of planetary exploration, the detection extra-terrestrial life forms, may not be realized if viable earth-type organisms are carried on non-sterile craft. Total mission failure may result, however, if the performance of spacecraft systems or components is degraded by the decontamination process.

The procedure developed for sterilizing spacecraft is accomplished in two steps. The first is a decontamination procedure to lower surface micro-organism population by exposure to an ethylene oxide - Freon 12 sterilant gas; the second is exposure to dry heat sterilization cycling to kill the remaining population. In practice, gaseous decontamination is applied at the sub-assembly level and to the entire spacecraft after assembly. Thermal sterilization is accomplished on the completely assembled spacecraft.

Dry heat cycling and gaseous chemical sterilants are highly effective in achieving biological sterility but are also capable of producing marked changes in material properties. Experience with the Ranger lunar spacecraft showed that dry heat cycling can cause considerable damage to electronic equipment through degradation of polymer materials. Preliminary studies of ethylene oxide - Freon 12 gaseous decontamination performed in connection with the Surveyor Spacecraft program clearly demonstrated that some polymeric materials are adversely affected by this treatment.

The compatibility of system materials with sterilization procedures must be established to gain the optimum confidence in performance reliability of spacecraft systems. Only materials whose properties are not seriously affected by exposure to the 12 percent ethylene oxide-Freon 12 sterilant gas mixture followed by dry heat cycling can be used for sterilizable spacecraft.

The primary objective of this program was to provide information on the compatibility of commercial polymeric products and a select group of metallic materials with a 12 percent ethylene oxide - 88 percent Freon 12 gas mixture. Because of the two-step sterilization procedure, the compatibility of materials with sterilant gas alone has no practical meaning. Therefore, all products were dry heat cycled after gas exposure, and the effects of the combined decontamination - sterilization treatment determined.

The sterilant gas mixture is highly penetrative and can cause chemical and physical changes not only on the surface of materials, but throughout their entire mass. Sterilant gas retained by materials after the decontamination step, can react further during the long, dry heat sterilization step and cause additional loss of properties.

The mechanisms of interaction of sterilant gas with polymers can be divided into two categories: physical and chemical. The type of property-degradation produced by the two modes of attack would be expected to be different. It was the purpose of the mechanisms portion of the program to separate the two mechanisms and identify chemical modifications in polymer systems with the aid of infrared, chromatographic, and gas-sorption techniques.

To gain the maximum amount of useful information from the program, the screening and mechanisms studies were conducted as parallel efforts. The same polymer classes were studied concurrently so that information from both phases could be used to establish a clearer picture of material behavior, and guide the direction of subsequent testing.

B. LITERATURE SURVEY

To save testing time in the screening portion of the program, a search of the literature was made to establish the compatibility of as many materials as possible with the ethylene oxide - Freon 12 gas mixture.¹ Since a complete literature search of this field was made by

¹R. F. Rydelek, "Literature Review of the Compatibility of Commercial Materials with Ethylene Oxide - Freon 12 Sterilant Gas Mixture," HAC Ref. No. 2748.40/910, JPL Contract 951003, January 1965.

Hughes in 1961-1962² it was not necessary to examine the literature prior to 1962. Materials of primary interest in this survey were those appearing on the lists submitted by JPL for test.

A separate survey³ was made in conjunction with the mechanism study to obtain information on the reactions of ethylene oxide and Freon 12, including sorption characteristics, corrosive effects and susceptibility to polymerization when in contact with polymeric and metallic materials.

Analytical methods for detecting small amounts of ethylene oxide, Freon 12, and water absorbed in polymers and deposited on metallic surfaces were also investigated. The recent literature provided little compatibility data. The major portion of usable information appeared in material published prior to 1962 and in the reports generated in the experimental study performed as part of the Surveyor Sterilization Program.^{4, 5} Applicable information found in the literature survey is summarized below. (References 1 and 3 should be consulted for more detailed information.)

Ethylene oxide is a highly reactive agent capable of participating in a wide variety of reactions and of modifying the physical and chemical properties of many compounds. It is capable of producing marked changes in material properties by reacting directly with base material or with impurities included during the manufacturing process. Two types of reactions are of particular interest in this program: condensation and

²M. Willard, V.K. Entekin, "A Literature Review of the Physical, Chemical, and Biological Properties of Ethylene Oxide-Freon 12 and Its Compatibility with Materials and Components," Surveyor Sterilization Part II, HAC RS-283, March 1962.

³A. L. Landis, "Literature Review of Mechanisms of Interaction of Ethylene Oxide and Organic and Inorganic Materials," HAC Ref. No. 2748.40/878, JPL Contract 951003, November 1964.

⁴M. Willard, "Compatibility of Materials and Components with Heat and Ethylene Oxide-Freon 12," Surveyor Sterilization Part I, HAC RS-277, January 1962.

⁵M. Willard, "Further Compatibility Studies of Materials and Components with Ethylene Oxide-Freon 12 and Heat," Surveyor Sterilization Part III, HAC RS-292, July 1962.

polymerization. The condensation of ethylene oxide with another compound proceeds by attachment of the broken epoxy ring to an active hydrogen.

Polymerization, catalyzed by bases and other inorganic salts, forms compounds of low volatility, potentially hazardous to electrical, contact and thermal control surfaces. Ethylene oxide in the vapor phase is highly penetrative, therefore the permeability of materials is an important factor. Freon 12 has solvent properties and tends to produce swelling in some elastomers. The presence of two fluorine atoms on the Freon molecule, contributes to its high stability and precludes direct chemical reaction with materials under test conditions used in this program.

Although investigation of alternate surface sterilants was not a required task of this program, a short discussion of information from the literature search on this topic is given below.

The ideal surface decontaminant should have the following characteristics:⁶

1. Non-corrosive
2. Non-deleterious to materials
3. Penetrative
4. Residual easily removed
5. Rapid Action
6. Low toxicity
7. Non-flammable, non-explosive
8. Easily stored
9. Easily handled
10. Bactericidal, sporicidal, virucidal, and fungicidal at atmospheric pressure.

The 12 percent ethylene oxide - 88 percent Freon 12 mixture meets most of these requirements, but shows a deleterious effect on some materials, requires extended exposure time to achieve sterility, and has toxic properties. Also, the complete removability of residual gases from materials that have been subjected to sterilization procedures

⁶ L. C. Miner, "Gaseous Sterilization of Pharmaceutical and Hospital Supplies," American Journal of Hospital Pharmacy, Vol. 16, June 1959.

has not been demonstrated. In addition, the gas mixture must be humidified to achieve optimum kill. Water vapor may produce undesirable side effects on materials undergoing decontamination, or may promote undesirable ethylene oxide reactions.

Other Gases - Other vapor phase sterilants have been investigated (6,7,8)

Ozone (O_3); methylbromide (CH_3Br);

Chloropicrin (Cl_3CNO_2); propylene oxide; $CH_3CH_2CH_2O$,

Epichlorohydrin (CH_2OCHCH_2Cl); epibromohydrin (CH_2OCHCH_2Br)

B-propiolactone (OCH_2CH_2CO); and ethylenimine (CH_2NHCH_2)

Each of these compounds has serious drawbacks. Ozone, B-propiolactone, and chloropicrin, for example, can produce corrosion or materials attack. Propylene oxide offers no advantages over ethylene oxide; epichlorohydrin and epibromohydrin are slightly more active than ethylene oxide, but have high boiling points and cannot be conveniently used in the vapor state. Methyl bromide shows considerably lower activity than does ethylene oxide. In general, none of the above compounds demonstrate sufficient advantages over ETO to warrant use as a sterilant. They may, however, have some value as additives for accelerating the activity or modifying the physical properties of the ETO-Freon 12 gas mixture.

C. PROGRAM PLAN

Most of the materials selected by JPL for evaluation in this program were those adhesives, encapsulants, laminates, lubricants, and films that had been previously used in spacecraft fabrication, primarily in the Ranger and Mariner programs, and had proven their ability to meet the other demands of spacecraft service. In addition, candidate materials were exposed to a preliminary heat screening at JPL. Only

⁷J. B. Opfell, "A General Review of Chemical Sterilization in Space Research," Preprint of paper presented at COSPAR Fourth Int'l Space Symposium, Warsaw, Poland, 1963.

⁸W. L. Beeers, M. Roha, Propriolactone as a Sterilant, OTS:P13139, 748, U.S. Dept. of Commerce, Office of Technical Services, 1959.

products which passed the heat screening were to be selected for evaluation with ethylene oxide - Freon 12 sterilant gas. Because of program time limitations, however, it was necessary to start gas screening of some materials before their compatibility with heat cycling could be established.

Commercial polymeric products are compounded of many ingredients. Plasticizers, curing agents, stabilizers, and pigments are added to base polymers to give them desired properties. To determine the role of additives in the ETO gas - material interaction, the mechanism study portion of the program examined both pure polymers, and commercial polymer products. To insure that the polymers studied were of high purity, a number of them were synthesized in-house. Details of the syntheses used are given in the appendix of this report. To gain the maximum amount of useful information from the program, the screening and mechanism portions were conducted as parallel efforts. The same polymer classes were studied concurrently so that information from both phases could be used to establish a clearer picture of material behavior and guide the direction of testing. Because the preponderance of products to be screened were epoxy and silicone polymers, major effort was directed toward the study of these polymer families.

In conjunction with the mechanisms study, representative polymers were subjected to dry heat cycling before they were exposed to ethylene oxide - Freon 12 to provide information on the effects of other sterilization procedures. This work also provided insight into the possibility of "passivating" a material with heat so that the degrading effects of subsequent gas exposure might be lessened. Heat tends to drive off more volatile additives and contaminants. Because these materials are potentially more reactive with ethylene oxide than is the base polymer, an overall benefit should result from a preliminary heat cycle.

Because of the need to monitor changes in sterilant gas composition during material exposure, development of suitable analytical techniques was an important task of this program.

II. EXPERIMENTAL

A. TEST APPROACH

The major portion of commercial polymers screening was accomplished by exposing test materials to a humidified 12 percent ethylene oxide - 88 percent Freon 12 gas mixture for 24 hours at 74°F followed by 24 hours at 104°F. Specimens were then subjected to three 36 to 40-hour heat exposures at 145°C under an atmosphere of dry nitrogen. Total sterilant gas exposure time for a material, then was 48 hours, and total time at temperature was 108 to 120 hours. Test specimens required approximately two hours to reach 145°C after they had been inserted into the heat cycling oven, therefore, the total time at elevated temperature exceeded the 108-120 figure by approximately five hours.

The room temperature and elevated temperature ethylene oxide-Freon 12 exposures were those defined in JPL Specification GMO-50198-ETS. The dry heat cycle corresponds to that given in JPL Specification XSO-30275 TSTA.

Material properties were measured before gas exposure (control specimens), after the two cycles of gas exposure, and after gas exposure, and dry heat cycling. (See Figure 1.)

A limited number of materials were exposed to dry heat cycling first and then to ethylene oxide - Freon 12 sterilant gas. The exposure conditions for both sterilant gas and heat cycling correspond to those given above. In the heat-followed-by-gas regime, material properties were measured after dry heat cycling and after the two cycles of gas exposure. In some cases, additional measurements were made after the room temperature gas exposure.

Three coupons of a material comprised a test set for non-destructive tests such as weight, volume, and hardness measurements, and each test set was processed through all stages of exposure.

Triplicate sets of coupons were prepared for destructive tests such as tensile and shear strength measurements. In the gas-followed-by-heat regime, one set was tested prior to gas exposure, the second after gas exposure, and the third after gas and heat exposure.

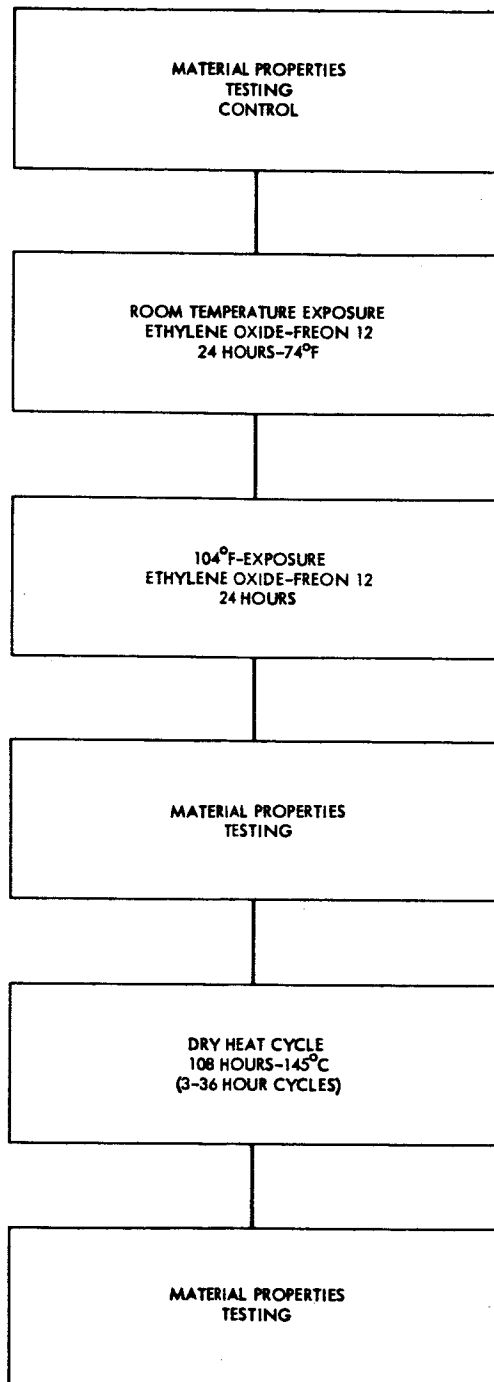


Figure 1. Exposure and test sequence.

For the major portion of the program, one test specimen of each material was used for volume resistivity measurements. Preliminary testing showed that variations in resistivity values from specimen to specimen were less than the variation inherent in the test method. Electrical test specimens of air-curing silicone adhesives RTV 108, RTV 140, and RTV 891 required special fabrication techniques which are detailed in Appendix A of this report.

The physical, electrical, and mechanical test methods used in this program are given in Table 1. It should be noted that it was not the purpose of the screening program to produce engineering data for the materials examined, but to establish their compatibility with the sterilization procedure. Although the major portion of the data was obtained through standard test methods, it was not possible to use these methods in all instances. Other test techniques were used, where noted, to provide information on relative changes in properties, rather than absolute values.

In the mechanism portion of the program test materials were exposed to sterilant gas and then analyzed to determine physical and chemical changes. Test specimen weight changes, and sterilant gas composition changes were monitored during gas exposure and subsequent vacuum exposure. The amount of sterilant retained by test materials after gas and vacuum exposure served to indicate whether the predominant mechanism involved in the gas - material interaction was physical adsorption (reversible) or chemical absorption (irreversible by vacuum exposure).

Infrared spectroscopy was used to study basic changes in polymer structure.

B. MATERIALS PROCUREMENT

The commercial polymeric products tested in the screening program were ordered directly from the manufacturer or local supplier to insure "freshness". Polymeric materials arriving in the fluid and uncured state were fabricated into sheet or other configurations suitable

Product Category	Test Method	<u>Key</u>	
Adhesives	Weight and Volume	<u>Weight</u>	= <u>Weight change</u>
	ASTM D 676		
	ASTM D1002	<u>Volume</u>	= Volume change by water displacement
Elastomers	Weight and Volume		
	ASTM D 257		
	ASTM D 412	<u>ASTM D 149</u>	= Kinematic viscosity
	ASTM D 676		
Encapsulants	Weight and Volume	<u>ASTM D 257</u>	= Volume resistivity
	ASTM D 257		
	ASTM D 412	<u>ASTM D 412</u>	= Tensile properties (Elastomers)
	ASTM D 638		
	ASTM D 676		
	ASTM D1706	<u>ASTM D 445</u>	= Viscosity (Lubricants)
Films	Weight		
	ASTM D 257	<u>ASTM D 638</u>	= Tensile properties (Plastics)
	ASTM D 882		
Lubricants	Weight	<u>ASTM D 676</u>	= Hardness (Elastomers)
	ASTM D 149	<u>ASTM D 790</u>	= Flexural Properties
	ASTM D 445	<u>ASTM D 882</u>	= Tensile properties (Films)
Plastic Laminates	Weight and Volume		
	ASTM D 257	<u>ASTM D1002</u>	= Shear Strength
	ASTM D 638		
	ASTM D 790	<u>ASTM D1706</u>	= Hardness (Plastics)
	ASTM 1706		

Table 1. Screening test methods.

for testing. Data on curing agent concentrations and detailed information on formulation techniques are given in Appendix A.

The materials studied in the mechanism portion of the program were formulated from pure polymers synthesized in-house or purchased from outside sources. Compounds of known composition were used to cure the polymers. Detailed information on formulations and synthesis techniques are given in Appendix B.

C. TEST EQUIPMENT

The vacuum oven shown in Figure 2 was used to accomplish dry heat cycling. The oven is capable of maintaining a vacuum of 29.5 inches of mercury for extended periods. The oven configuration made the use of thermocouples difficult, therefore test temperatures were established as follows: A thermometer was placed at the center of the oven and temperature observed as the oven temperature control settings were increased; when the temperature stabilized at 145°C, the oven was loaded with materials to simulate a dry heat cycle. A thermometer was placed between sheets of test material at the center of the oven, and the time required to reach 145°C noted.

Figure 3 is a schematic diagram of the ethylene oxide - Freon 12 exposure apparatus shown in Figure 4. The exposure chamber (1/2 inch steel walls) was wrapped with heating tapes and insulated to provide temperature control for the sterilant gas exposure at 104°F. Temperature was measured with a shielded thermocouple extending into the chamber.

The apparatus shown in Figure 5 was fabricated to measure the rate of ethylene oxide sorption by pure polymers and permit degassing of test samples. The all glass apparatus consisted of a manifold, valve system, sample container, manometers, and cold trap. A mercury diffusion pump was used to attain pressures in the range 10^{-4} to 10^{-5} mm Hg.

A Beckman GC-2 gas chromatograph was used for analysis of system gases. A helium carrier gas pressure of 20 psi and thermal conductivity detector filament current of 200 ma were sufficient to give good resolution with a 12 foot dioctyl isosebacate column modified with 12 percent Ucon 50 HB-2000. Ethylene oxide elution time is four minutes, and Freon 12

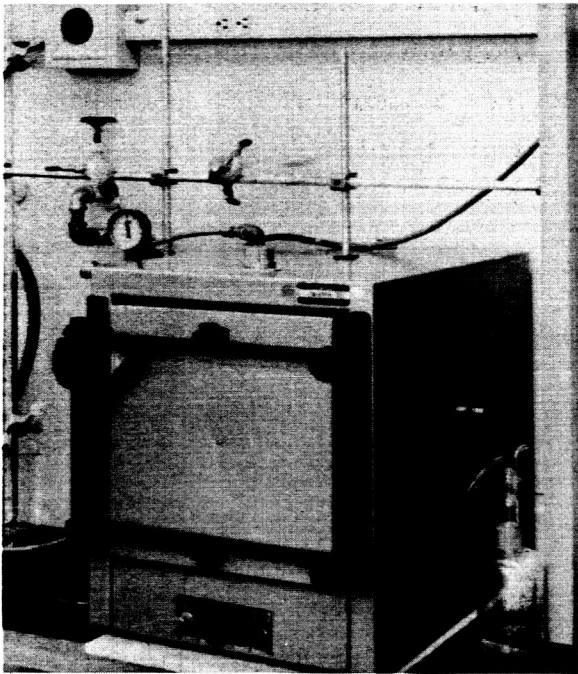


Figure 2. Vacuum oven used for dry heat cycling.
(HAC Photo No. R103537)

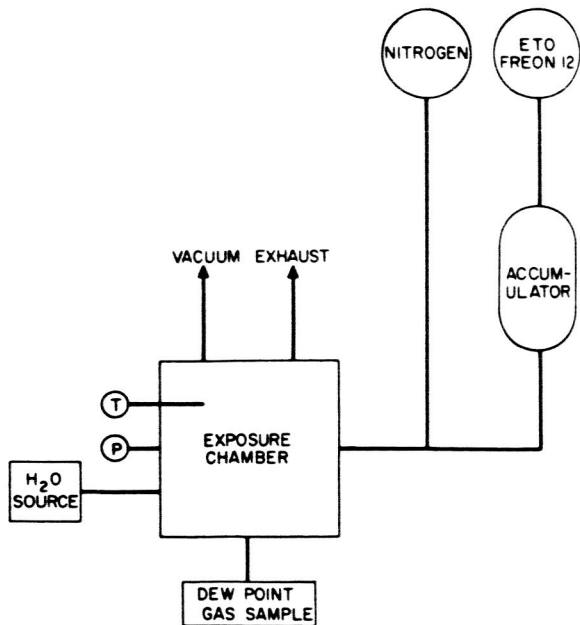


Figure 3. Ethylene oxide-Freon 12 exposure apparatus schematic.

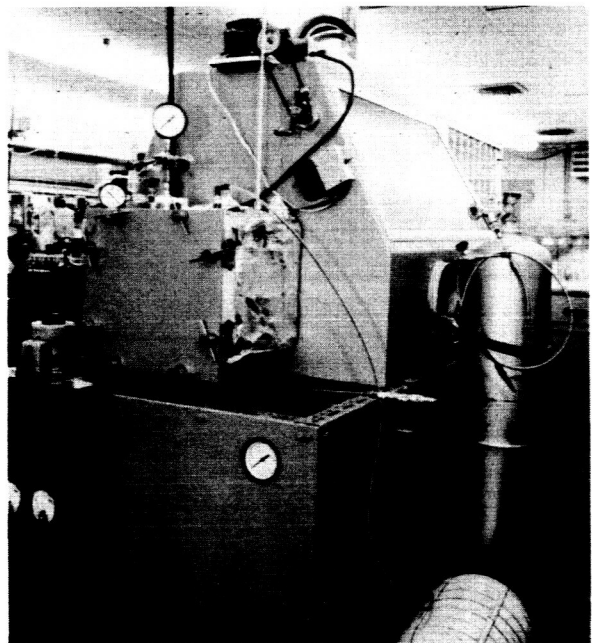


Figure 4. ETO-Freon 12 exposure apparatus.
(HAC Photo No. R103538)

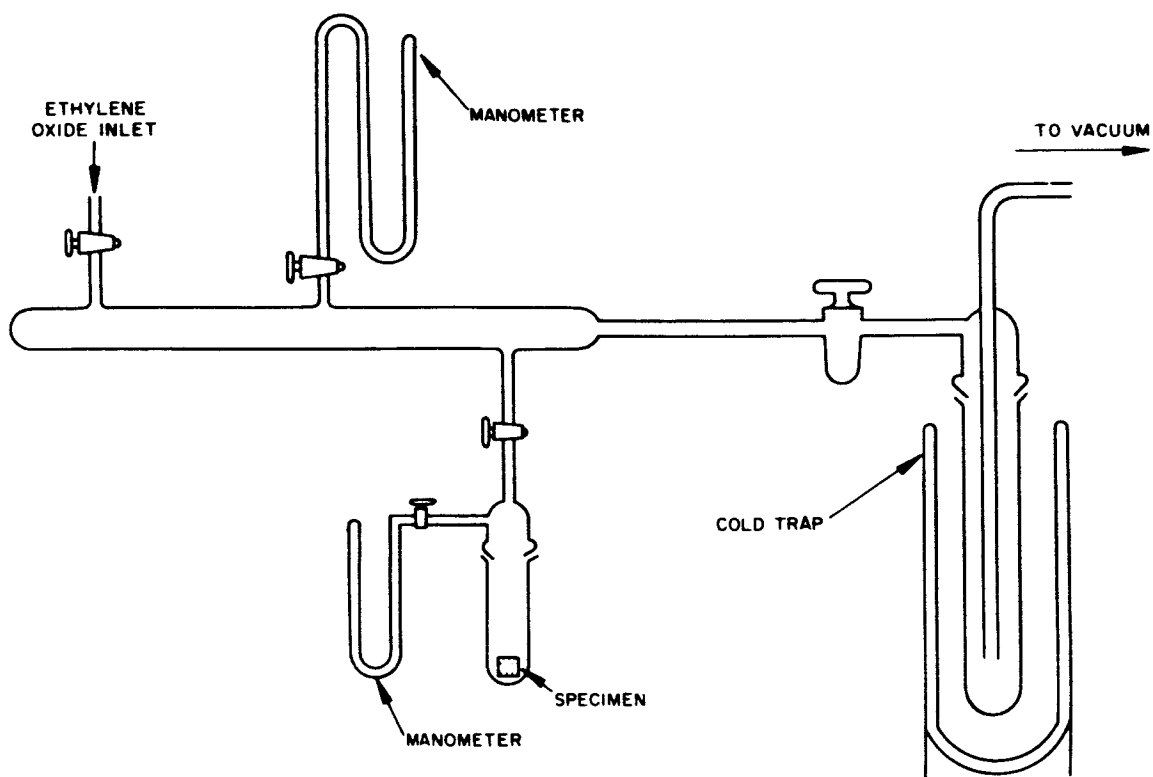


Figure 5. Ethylene oxide sorption apparatus diagram.

is 1.3 minutes with the column at 50°C. A Beckman IR-5 infrared spectrophotometer was used to obtain spectra of pure polymers before and after exposure to ethylene oxide. A Beckman 10 meter variable path gas cell was used in conjunction with the IR-5 to measure ETO and Freon 12 concentration during the sorption studies.

D. TEST PROCEDURES

1. Dry Heat Cycling

Test specimens were placed on metal oven shelves with sufficient spacing to allow free flow of nitrogen around them. The chamber was evacuated, filled with dry, high purity nitrogen, and then evacuated again. After bringing the oven to atmospheric pressure with nitrogen, a low gas flow was established and maintained during the remainder of the test.

At the end of 36 to 40 hours at test temperature, materials were removed from the oven, allowed to stabilize at room temperature, and visually examined. The above procedures were repeated three times, subjecting the test specimens to temperature for a minimum of 108 hours.

2. Ethylene Oxide-Freon 12 Gas Exposure (see Figure 4)

Sheets of elastomers and other non-rigid materials were suspended from the top of the exposure chamber, with a minimum space of 1 inch between them to allow circulation of gas. Small, rigid coupons were arranged around the rim of glass petri dishes and placed on the bottom of the chamber (see Figure 6).

The exposure chamber was evacuated and maintained at 29.5 in Hg for 20 minutes. Ethylene oxide-Freon 12 mixture was slowly bled from the storage cylinder into the evacuated accumulator where it was allowed to temperature stabilize at 10 psig. The evacuated chamber was then opened to a heated water source, until water vapor pressure in the system reached 20 mm Hg. Gas was bled from the accumulator into the exposure chamber until atmospheric pressure was obtained.

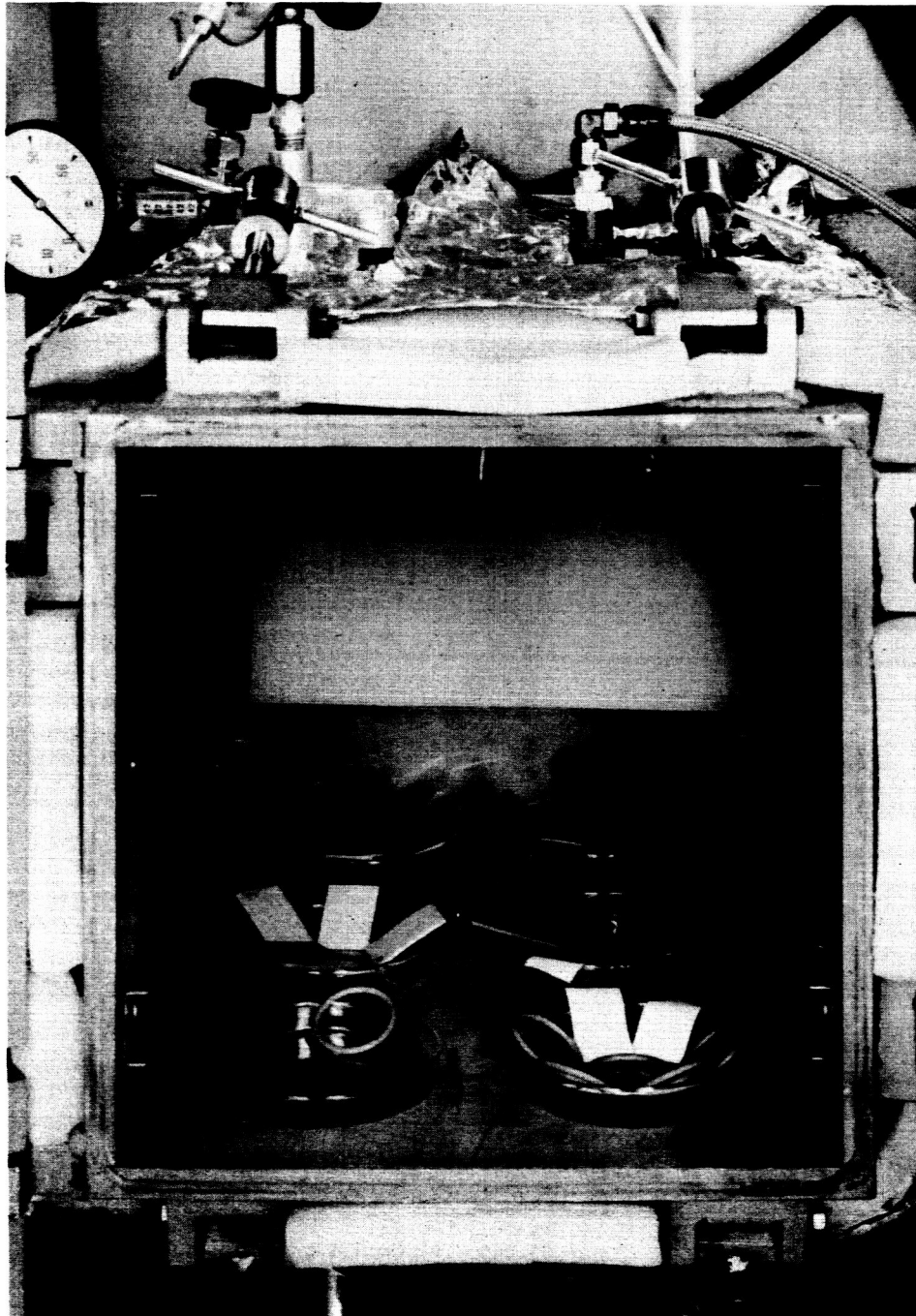


Figure 6. Exposure chamber and samples.
(HAC Photo No. R 103539)

Analysis of gas in the exposure chamber was performed with the gas chromatograph. At the end of the 24 hour exposure period, the chamber was flushed with dry nitrogen, evacuated to 29.8 in Hg for 30 minutes and brought to atmospheric pressure with air.

The procedure for exposure at 104°F is identical to that described above. The chamber is heated for a period of three hours prior to insertion of test specimens which were allowed to stabilize at chamber temperature for one hour before gas was admitted.

3. Dew Point Measurement

The dew point device was evacuated and then opened to the exposure chamber through a valve assembly. The humidity of the gas trapped in the device was determined by adding small pieces of ice to the water in the thermometer well, and stirring vigorously. The temperature at which a haze, denoting water condensation, was observed was taken as the dew point. Dew points in the range 40 to 55°F indicate proper chamber humidity for room temperature (74°F) gas exposure and 66°F to 78°F for exposure at 104°F.

4. Sorption Studies

Specimens of test material were placed in the apparatus and degassed by exposure to vacuum. The apparatus was then back filled with sterilant gas, and the sorption rate obtained by observing the mercury level in the manometer. The gas pressure in the test chamber was maintained close to atmospheric by admitting gas as needed. Test specimens were periodically removed from the apparatus and weighed until no increase in weight was detected. Unreacted sterilant was removed from the test material by the application of vacuum, and trapped in a liquid nitrogen trap for subsequent infrared analysis.

5. Infrared Studies

Thin cast films of pure polymer were exposed to ethylene oxide as described above. Infrared spectra of both exposed and unexposed polymer were obtained with the Beckman IR-5 spectrophotometer.

III. DISCUSSION

A. RESULTS AND DISCUSSION

Tables 2 through 8 give changes in physical, mechanical, and electrical properties of commercial products exposed to ethylene oxide-Freon 12 sterilant gas and dry heat cycling. Tables 8 through 13 give compatibility ratings for the same products with the exception of coating materials which are not rated.

Ratings are based on net changes produced by both gas and dry heat exposure. Material properties measured after gas exposure alone were not used for rating and are presented only to give a better picture of overall material behavior. Criteria used for product ratings were established by JPL and are discussed below according to functional category.

The data in the tables should be interpreted in light of the following facts:

1. Data reflects the behavior of a specific sample of a commercial product. Because products are subject to considerable batch variations in additives, contaminants, curing agents, cure conditions, and storage, it is possible that another sample of the same product might show a different compatibility rating.
2. Data give a relative indication of material behavior and are not intended for design purposes. The form in which a material was tested was not necessarily the form in which it is ordinarily used. Determination of mechanical properties of encapsulants such as Stycast 1095, and Sylgard 182, required that they be cast into tensile specimens. The surface area to volume ratio was probably much greater than would occur in spacecraft components.
3. Electrical property values are a function of the measuring techniques and apparatus used, and the surface condition of the test specimen. Small variations in surface texture can produce large variations in value. Although ASTM D257 was followed, the volume resistivity values presented in this report

are to be used only as indications of material behavior and not as absolute values. Because electrical measurements were made on the same specimen as it progressed through the stage of exposure, the relative change in volume resistivities can be used with a high degree of confidence for evaluating material behavior.

4. Compatibility ratings presented in this report apply to materials which have been exposed to the sterilization procedure defined in the Test Approach. Exposure to longer gas and heat cycles or to multiple combinations of gas and heat cycles would undoubtedly produce greater property changes.

Adhesives

The following criteria, established by JPL, were used to rate adhesives:

Compatible (C)

1. Materials retained 80 percent or more of their original tensile shear strength
2. Weight loss less than one percent.

Marginal (M)

1. Materials retained between 70 and 80 percent of their original tensile strength
2. Weight loss was one to four percent.

Not Compatible (N. C.)

1. Materials retained less than 70 percent of their original tensile strength
2. Weight loss was greater than four percent

Of the eleven adhesives tested, two rated as marginal because of weight loss. Although Eccobond 55/11 was not rated because weight change data was lacking, minor changes in shear strength indicate that it is compatible. No material was rated as non-compatible. Discoloration and blistering were evident on cast buttons of E Solder 3022 and Eccobond 57/C used for weight and volume measurements. Discoloration appeared during

sterilant gas exposure and indicates a chemical reaction. Mechanical test data did not indicate loss of properties, however, the shear test specimens used, presented only a small area of adhesive for attack. It is possible that if more adhesive were exposed, greater losses in shear strength would occur. Five materials showed an increase in shear strength after heat cycling due to a post cure effect.

Coatings

No compatibility ratings have been given to coatings, however, abrasion resistance data in Table 3 gives an indication of material behavior. The Taber abrasion test method was selected because it indicates changes in several parameters: material integrity, hardness change, and adhesion characteristics. A CS-10 Calibrase wheel was used under a 1000 gram load for all abrasion testing.

Test specimens of Alkanex 9522, Pyre ML, and Electrofilm 4306 were prepared by spraying, and Eccocoat IC 2 by brushing onto prepared aluminum plates.

Table 3 shows that Electrofilm 4306 and Pyre-ML both gained in abrasion resistance as a result of gas exposure. Pyre-ML experienced a 16 percent gain after heat exposure and Electrofilm a one percent gain. Alkanex 9522, was unaffected by ETO-Freon 12, but showed a 15 percent loss after heat cycling.

Samples of Eccocoat IC 2, polyurethane, Emerson Cuming, were prepared and abrasion tested, however, the results were inconclusive and the test data is not presented in this report. Eccocoat is a highly resilient material, and did not lend itself to abrasion testing under the conditions used to test the other coating materials. Several thousand abrasion cycles caused no appreciable wear on the samples tested. Control, Gas, and Gas and Heat specimens all gained weight during abrasion testing, indicating that the coating stayed intact and picked up grit during the test. Although there is no specific data to rate Eccocoat IC 2 by, general behavior and appearance indicates that it is compatible with both stages of the sterilization procedure.

Elastomers

Criteria used for rating elastomers were:

Compatible:

1. Retained 80 percent or more of original tensile strength.
2. Retained 80 percent or more of original percent elongation.
(Elongation measurements were not made on RTV elastomers.)
3. Hardness change less than 6 units.
4. Weight loss less than one percent.
5. Volume resistivity drop of less than one order of magnitude.

Marginal:

1. Retained 70 to 80 percent of original tensile strength.
2. Retained 70 to 80 percent of original percent elongation.
3. Hardness change was more than 6 units but less than 10.
4. Weight loss was between one to four percent.

Not Compatible:

1. Retained less than 70 percent of original tensile strength.
2. Retained less than 70 percent of original percent elongation.
3. Hardness change was more than 10 units.
4. Weight loss was greater than four percent.

Of the 18 elastomers screened, nine qualified as marginal and one as not compatible. PMP 6100, silicone, lost 27 percent of its original tensile strength and 16 percent in elongation. Parker Seal compound S 417-7, silicone, lost 60 percent elongation, but only 3.5 percent in tensile strength when tested as an "O" ring. Six compounds were rated as marginal because of hardness change alone. These compounds, however, came within one or two points of meeting the acceptable hardness change set for a compatible material.

Twelve elastomers increased in hardness after gas exposure, and ten of these showed further increases during heat cycling. The change in properties was probably produced by heat and vacuum during the 104⁰F exposure, and by heat during the heat cycle. Chemical attack by sterilant gas did not appear to play a major role.

Changes in tensile strength of a number of elastomers and elastomeric encapsulants are plotted in Figure 7. Gas exposure increased tensile strength in five materials, of which four showed tensile losses after heat cycling. Eight materials lost tensile strength during gas exposure, six of which showed further losses after dry heat cycling; two increased in strength. No conclusions can be drawn concerning relative compatibilities of the polymer families. Of the two fluorocarbons tested, VITONS B 60 and 90, one showed a comparatively large net increase and the other a large decrease in properties. Silicones and fluoro-silicones also showed a spread in tensile change values.

Data in Table 4 shows that material degradation proceeded along several different lines:

1. Chemical attack by gas
2. Heat degradation
3. Physical adsorption
4. Chemical attack by gas enhanced by subsequent heat cycle.
4. Desorption (gain in properties) by subsequent heat cycle.

Parker Seal compound S417-7 showed a major loss of properties after gas exposure and a relatively minor increased loss after heat cycling. Chemical attack was the major degradation mechanism here.

PMP6100, and Parker Seal Compound L-449-6/60, were not appreciably changed by gas exposure, and showed a major loss in tensile properties only after heat cycling.

VITON B90 lost tensile strength after gas, but regained the loss after heat cycling. This indicates that gas was physically adsorbed, and then desorbed by heat.

The "chemical attack enhanced by heat cycling" effect no doubt occurred in a number of products, however, specific examples would be difficult to select from program test data. The effect of heat, and of gas and heat can only be determined by establishing property changes by heat alone; this was not a required task of this program.

Volume resistivities of the elastomers tested remained relatively stable with eight materials showing an increase in value. Where decreases did occur, they were less than an order of magnitude.

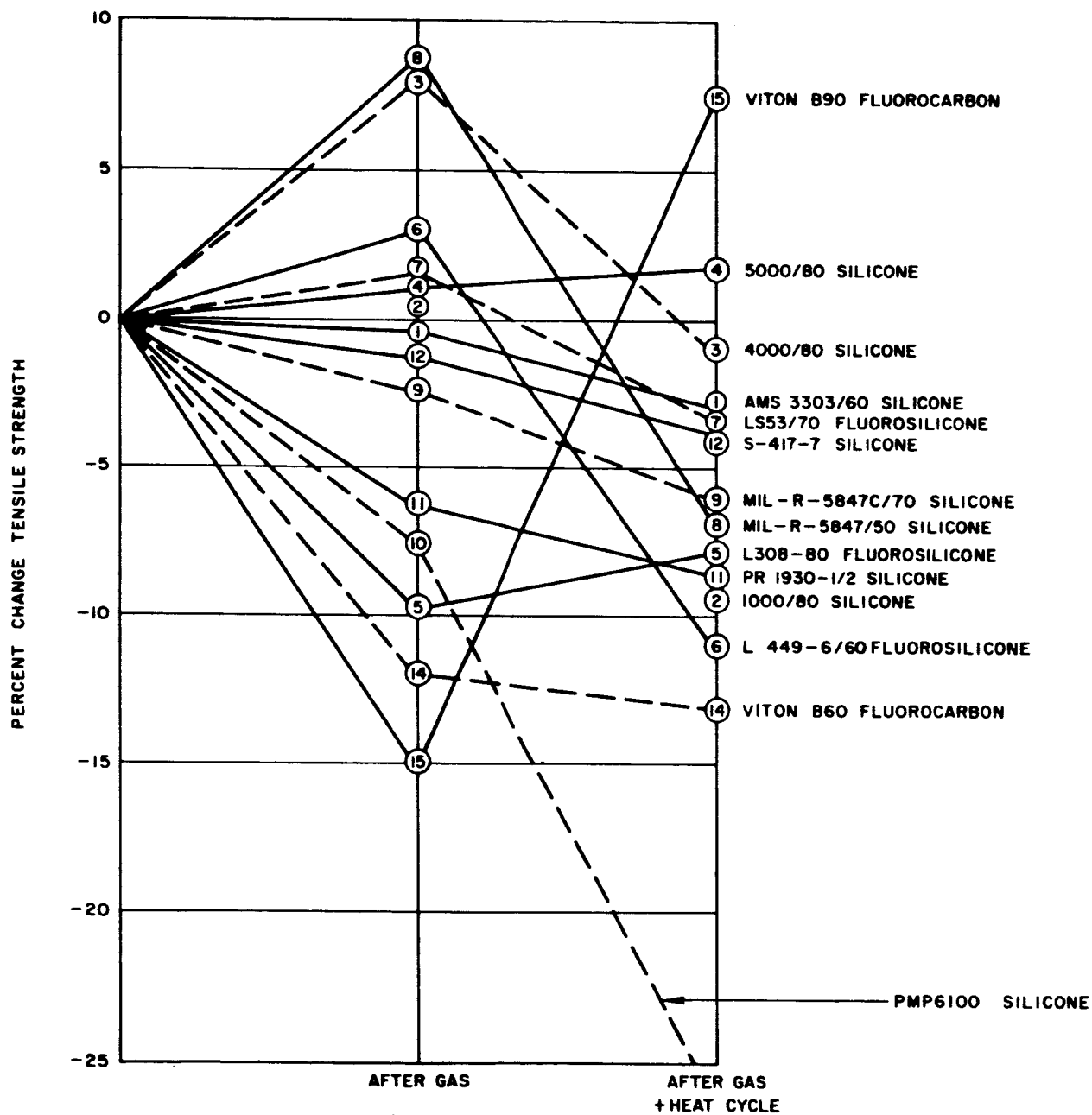


Figure 7. Changes in tensile strength of elastomers and elastomeric encapsulants.

The behavior of pure silicone polymers was studied in the mechanisms portions of the program discussed in Section C.

Encapsulants

Criteria used to rate encapsulants are presented below.

Compatible:

1. Weight loss less than one percent.
 2. Volume shrinkage less than four percent.
 3. Hardness loss less than ten units.
- Loss of volume resistivity less than an order of magnitude.

Marginal:

1. Weight loss between one and four percent.
2. Volume shrinkage was four to six percent.
3. Hardness loss between 10 and 15 units.

None of the materials tested were rated not compatible. All of the encapsulant materials tested arrived in the liquid and uncured state, and were cast into sheet form to facilitate testing. The Stycast materials presented problems; it was difficult to obtain a sheet of homogeneous texture, although several different mixing and casting techniques were tried. Shear specimens were prepared with Stycast 2651 after several attempts failed to produce a suitable sheet of this material. Although mechanical data was not used to rate encapsulant materials, it was used as a measure of gas - material interaction.

Of the six products tested, three rated as marginal. RTV 11 lost 1.01 percent in weight. While this is only slightly above the permissible loss, tensile data shows that the overall integrity of the material was degraded. Stycast 1095 and Stycast 2651 both showed volume shrinkage of greater than four percent.

Sylgard 182 showed minor changes in physical properties, and a loss of 52 percent in tensile strength. This tensile loss may not be a valid figure because the material has poor tear resistance, and standard tensile testing techniques may not be applicable. This is an indication of degradation, however, and its use should be confined to applications where mechanical strength is not a requirement.

None of the materials tested showed an appreciable drop in volume resistivity.

Films

Criteria used to rate polymer films are given below

Compatible:

1. Retained 80 percent of original tensile strength and elongation.
2. Weight loss was less than one percent.
3. Drop in volume resistivity was less than an order of magnitude.

Marginal:

1. Retained 70 percent to 80 percent original mechanical properties.
2. Weight loss was one to four percent.

No material tested rated as non-compatible. Of the five products tested, only Mylar Type D, 3 mil, rated as marginal.

Data in Table 6 show that films properties changed drastically after gas exposure and again after dry heat cycling. These drastic changes demonstrate that material behavior is not only a function of material composition, but also of configuration. The films tested in this program ranged in thickness from 2 mil to 5 mil, and presented an ideal configuration for gas-material interaction because of high surface to volume ratio, allowing the sterilant gas to diffuse through the entire film. The effect of thickness is also demonstrated by the difference in behavior of 3 mil and 5 mil Mylar Type D. Although they are the same in chemical structure, the 3 mil material showed much greater losses in weight and mechanical properties, as a result of gas exposure and heat cycling. The major tensile loss in the 3 mil material occurred during the heat cycle. Mylar 65 H. S., showed an 82 percent increase in tensile strength after gas and heat exposure. The material is heat shrinkable, and evidently additional shrinkage occurred in exposure, resulting in a gain in tensile properties.

H-film lost 50 percent of its tensile strength and Tedlar 200 lost 25 percent during the gas exposure. Both materials regained some of the loss when they were heat cycled, indicating that a reversible physical

adsorption of gas had taken place. Although both materials qualify as compatible by the criteria used to rate them, additional testing should be performed to establish their use-limitations.

An H-Film sorption study was performed in the mechanisms portion of the program. A sample was equilibrated with the sterilant gas mixture, and the residual gas above the sample analyzed. The analysis showed that most of the ethylene oxide had been scavenged by the test specimen, leaving an atmosphere of Freon 12. Exposing the specimen to a vacuum did not remove the sorbed ethylene oxide. The mechanism of the gas-material interaction is not clear. H-Film, polyimide, does not have appreciable active hydrogens in its molecular structure. Because ethylene oxide attack proceeds through an active hydrogen it is doubtful that a chemical interaction occurs. Degradation may occur by gas molecules entering the structure of the material and forcing polymer chains apart and weakening internal forces.

Reinforced and Unreinforced Plastics

Criteria used to rate reinforced and unreinforced plastics are given below.

Compatible:

1. Retained more than 80 percent tensile strength.
2. Hardness change was less than eight units.
3. Weight loss was less than one percent.
4. Volume resisitivity did not drop by an order of magnitude from original.

Marginal:

1. Retained 70 to 80 percent tensile strength.
2. Hardness change was 8 to 12 units.
3. Weight loss was one to four percent.

No material tested rated as not compatible; of the fourteen products tested, three were rated as marginal. Fiber Glass 91 LD, Laminate NS, and Micarta LE-221 showed greater than one percent weight losses.

Laminate EG 752 showed a volume shrinkage of 6.7 percent, however, volume change was not used to rate materials in this category.

Diall FS-4 lost 18 percent of its tensile strength, Diall FS-10 lost 17 percent and Laminate EG 752 lost 14 percent. These tensile losses were not large enough to qualify the products as marginal, however, they demonstrate that appreciable material degradation occurred during heat cycling. Changes in tensile strength for a number of laminate materials are plotted in Figure 8. All of the materials shown experienced some change as a result of gas exposure. Gas exposure increased tensile strength of six products, and subsequent heat cycling further increased tensile strength of three of these materials. Five materials lost tensile properties during gas exposure, four of which show further losses after the subsequent heat cycle. In general, phenolic materials show the least net change in tensile properties, epoxies show moderate changes and diallylphthalate materials show the greatest change.

Volume resistivity measurements on Laminate EG 758 T showed excessive spread and are not reported in Table 7. The material is copper clad and metal particles were embedded on the edges of the laminate when test samples were sawed from sheet stock. The metal particles provided a current path across the polymer layer and produced unreliable data. In general, neither gas nor heat produced appreciable changes in the volume resistivity of test materials. Diall FS-10 and Laminate NS showed a drop in volume resistivity after gas exposure and an increase after heat cycling.

Phenolic and epoxy polymers were examined in the mechanisms portion of the program and are discussed in the Mechanism Section of this report.

Lubricants

Three materials were tested in this category and data is presented in Table 8. The term lubricant is a misnomer for the Dow Corning 5 compound. It is primarily used as a dielectric grease to reduce current leakage of insulators, and as a moisture repellent seal for electronic components, therefore no mechanical testing was performed on this product.

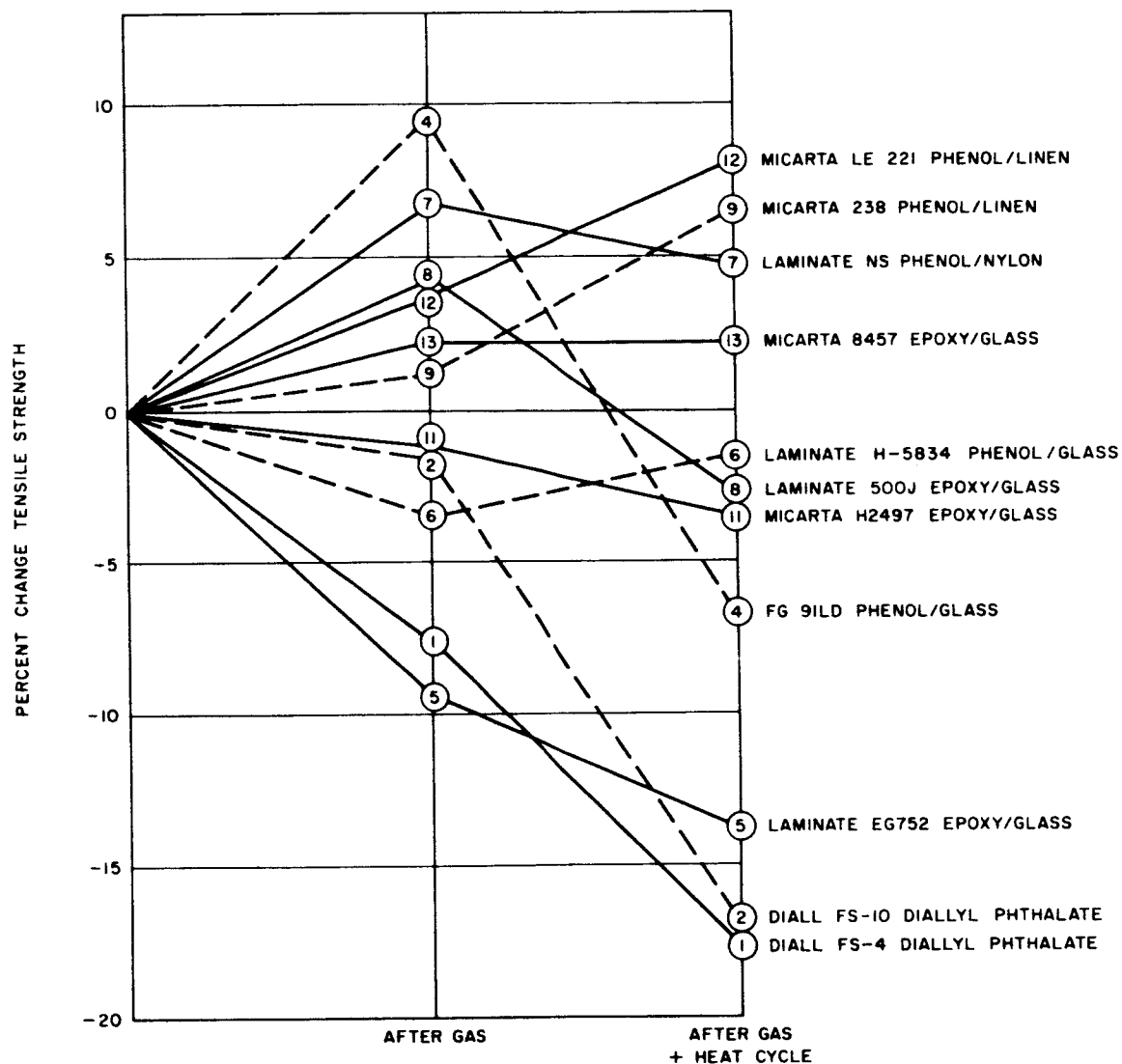


Figure 8. Changes in tensile strength of reinforced and unreinforced plastic laminates.

Electrical properties of the lubricants were tested in a Dow Corning Ball Cell according to ASTM Method D-149, "Dielectric Break-down Voltage and Dielectric Strength of Electrical Insulating Materials", using a 50 mil gap. All voltages in Table 8 are direct readings and have not been reduced to KV per mil. Although electrical data was not used to rate lubricants they are presented here to give a better picture of material behavior.

The F-50 fluid, as received, showed an abnormally low break-down voltage, possibly due to contamination. The fluid was exposed to vacuum for ten minutes at 150°F to remove water or polar solvents. This pretreatment brought the dielectric breakdown voltage in line with values appearing in the manufacturer's literature.

Kinematic viscosities were measured with calibrated Cannon-Fenske tubes.

Criteria used to rate lubricants are:

Compatible:

1. Viscosity changes less than five units.
2. Weight losses less than one percent.

Marginal:

1. Viscosity changes between five and ten units.
2. Weight losses between one and two percent.

Not Compatible:

1. Viscosity changes more than ten units.
2. Weight losses greater than two percent.

DC 5 Grease, and GE F-50 fluid are rated compatible, and DC 200 Silicone Fluid as not compatible because of a large viscosity change.

Gas exposure caused a 15.8 centistoke drop in GE F-50 viscosity, however, subsequent heat cycling reversed the trend and produced a final value within 1.5 centistokes of the original. A reversible physical adsorption of sterilant gas is indicated by this behavior. It is interesting to note that a small amount of adsorbed gas (less than 0.01 percent weight change) produced a large viscosity change in the F-50 fluid.

Other Products

Products Submitted by JPL for Test. Minor testing was performed on specimens of commercial materials submitted by JPL for screening which do not appear on the original JPL products lists. Test results are discussed below.

1. G. E. PPO, PENTON, CELCON, POLYSULFONE, TEFLON
Table 14 shows weight, volume, and hardness changes which took place after gas, and gas and heat exposure. Although tensile properties were not determined, all materials appear to rate as compatible based on parameters measured.
2. HADBAR 9000-65, polyurethane. Specimens submitted by JPL for test were exposed to sterilant gas at 104°F for 30 hours, and dry heat cycled for 112 hours at 145°C and then tensile tested. A tensile strength of 3335 psi and a percent ultimate elongation of 630 were obtained. No control specimens were tensile tested. The appearance of the tensile test specimens did not change during heat and gas exposure; however, the compression set specimen swelled appreciably during heat exposure. Large bubbles were noted within the specimen after the first heat cycle.
3. Mystik Tape 7000G, Mystik Tape products, glass fabric-silicone adhesive. Peel tests, performed on this material, are summarized below:

<u>Tape Width</u>	<u>Exposure</u>	<u>Average P. S. Pounds</u>	<u>Peel Strength oz/inch</u>	<u>% Δ Peel</u>
0.490	Control	1.15	37.5	---
0.425	Gas	0.80	30.1	-20
0.435	Gas + Heat	1.21	44.6	+19

Gas exposure lowered the peel strength of the tape, however, subsequent heat cycling produced an overall increase. Other than a slight discoloration, no observable changes occurred as

a result of exposure to the sterilization regime, and Mystik Tape 7000 G can be considered compatible.

4. American Super Temp Wire, H-Film and Teflon FEP coated.

The wire was exposed to gas and to heat cycling and breakdown voltage measured; results are given below:

EXPOSURE	BREAKDOWN VOLTAGE
Control	19 KV
Gas	18.5 KV
Gas and Heat	17.5 KV

Weight change data was inconclusive because of the small sample size, however, it showed the wire gained after gas exposure, and returned to approximately the control value after heat cycling.

B. ANALYTICAL TECHNIQUES

JPL Spec GMO-50198-ETS requires that an ETO concentration of 500 ± 50 mg/liter be maintained in the sterilizer chamber during gas exposure.

At atmospheric pressure, the 12 percent ETO-88 percent Freon 12 mixture barely falls within this limit. Any air dilution at 104°F lowers the ETO concentration below the required concentration, therefore, frequent analysis of chamber concentration was required.

One of the objectives of this program was the selection of practical analytical methods for measuring ethylene oxide, Freon 12, water vapor, and air.

The ideal method should provide a simultaneous analysis of all constituents with a high degree of accuracy. Because the literature provided little information on suitable procedures for accomplishing this, considerable experimental effort was required to fulfill the program requirements. A brief summary of this effort is given below.

Past experience at Hughes showed that ethylene oxide, Freon 12, and air, can be analyzed chromatographically with accuracies of ± 0.1 percent using a dioctyl isosebacate column modified with 12 percent UCON HB - 2000.

Water, however presents difficulties because of its tendency to be strongly adsorbed on most column materials at operating temperatures required to give good separation of other gaseous constituents. Columns with teflon substrates have been generally used for water analysis because of the non-adsorptive characteristics of the fluorocarbons. Two teflon columns were investigated:

1. Carbowax 1540 on teflon
2. Polypropylene glycol on teflon

To gauge the ability of these columns to separate water vapor from a gas sample, they were first tried with a humidified nitrogen stream.

The Carbowax on teflon column produces a diffuse chromatogram peak, for water, and the polypropylene glycol on teflon column produced a well-defined peak at relative humidities of 50 to 70 percent.

Unfortunately, when tried with the ETO-Freon 12 mixture, these columns could not separate Freon 12 and air at the operating conditions required to produce a quantitative water peak.

It is doubtful that a single chromatographic column can be used for analysis of ethylene oxide, Freon 12, air and water in gas samples. Water analysis requires high column temperatures and carrier gas flows with short columns. These conditions are not favorable for good separation of air and Freon 12.

C. MECHANISM STUDY

Eight polymers were studied in the mechanism portion of the program. Major emphasis was on silicone and epoxy polymers because most of the commercial products screened in this program belonged to these families. In addition, a study was made of H-Film (polyimide) due to its unusual reaction to gas exposure.

Silicone Polymers. Four high purity silicone polymers were prepared for study. The compositions of formulations used are given below, and

detailed information on formulation techniques and cure cycles are given in Appendix B.

Silicone Formulation 1

polydimethylphenylmethoxysiloxane (C 1962-34)	100 parts
triethoxymethylsilane	8 parts
dibutyltin dilaurate	2 parts

Silicone Formulation 2

polydimethylsiloxane (C1090-61A)	100 parts
triethoxymethylsilane	8 parts
dibutyltin dilaurate	3 parts

Silicone Formulation 3

polydimethylsiloxane (C1090-60A)	100 parts
triethoxymethylsilane	8 parts
dibutyltin dilaurate	2 parts

Silicone Formulation 4

polydimethylsiloxane (C1090-60A)	100 parts
triethoxymethylsilane	8 parts
dibutyltin dilaurate	2 parts
powdered silica, 270 mesh	50 parts

Polydimethylsiloxanes are representative of silicone compounds generally used in the formulation of commercial products. The pure polymer (C1090-60A) was prepared by cohydrolysis of difunctional dimethyl silanes. A similar polymer (C1090-61A) was prepared by equilibration of cyclic dimethylsiloxanes with sulfuric acid.

Polydimethylphenylmethyl siloxane is also widely used in commercial formulations. The phenyl groups attached to the chain silicone atom impart greater thermal stability to the compound. The pure polymer (C1962-34) was prepared by cohydrolysis of dimethyldichlorosilane and phenylmethyldichlorosilane.

All of the above polymers were cured with triethoxymethyl silane using a dibutyltin dilaurate catalyst.

Sorption Studies

Specimens of test material were placed in the sorption apparatus and degassed by exposure to vacuum. The apparatus was then back filled with pure ethylene oxide, and the sorption rate obtained by observing the mercury level in the manometer. The ethylene oxide pressure in the test chamber was maintained close to atmospheric by admitting gas as needed. Test specimens were periodically removed from the apparatus and weighed until no increase in weight was detected. Unreacted ethylene oxide was removed from the test material by the application of vacuum. Data from the sorption studies are presented in Table 15.

All formulations adsorbed ethylene oxide in excess of two percent by weight, and formulation 2 took up 8.30 percent by weight. The small amount of gas retained by the polymers after vacuum exposure indicates that a physical rather than chemical gas-material interaction had occurred.

Infrared Studies. To determine if ethylene oxide exposure caused structural changes in the polymer, infrared spectra were obtained of both exposed and unexposed films of formulation 3 using a Beckman IR-5 spectrophotometer. Figure 9 shows typical spectra of treated and untreated polymers. No important structural changes are indicated.

Tables 16 and 17, give mechanical data for three commercial silicone elastomers which were exposed to (1) Gas first followed by dry heat cycling, and (2) Dry heat cycling first and then to gas. The objective of this effort was to determine if heat cycling passivated materials by driving off more volatile, hence more reactive constituents before gas exposure, and produced less overall change in properties. The data shows that in the case of the three representative materials, little net difference occurred.

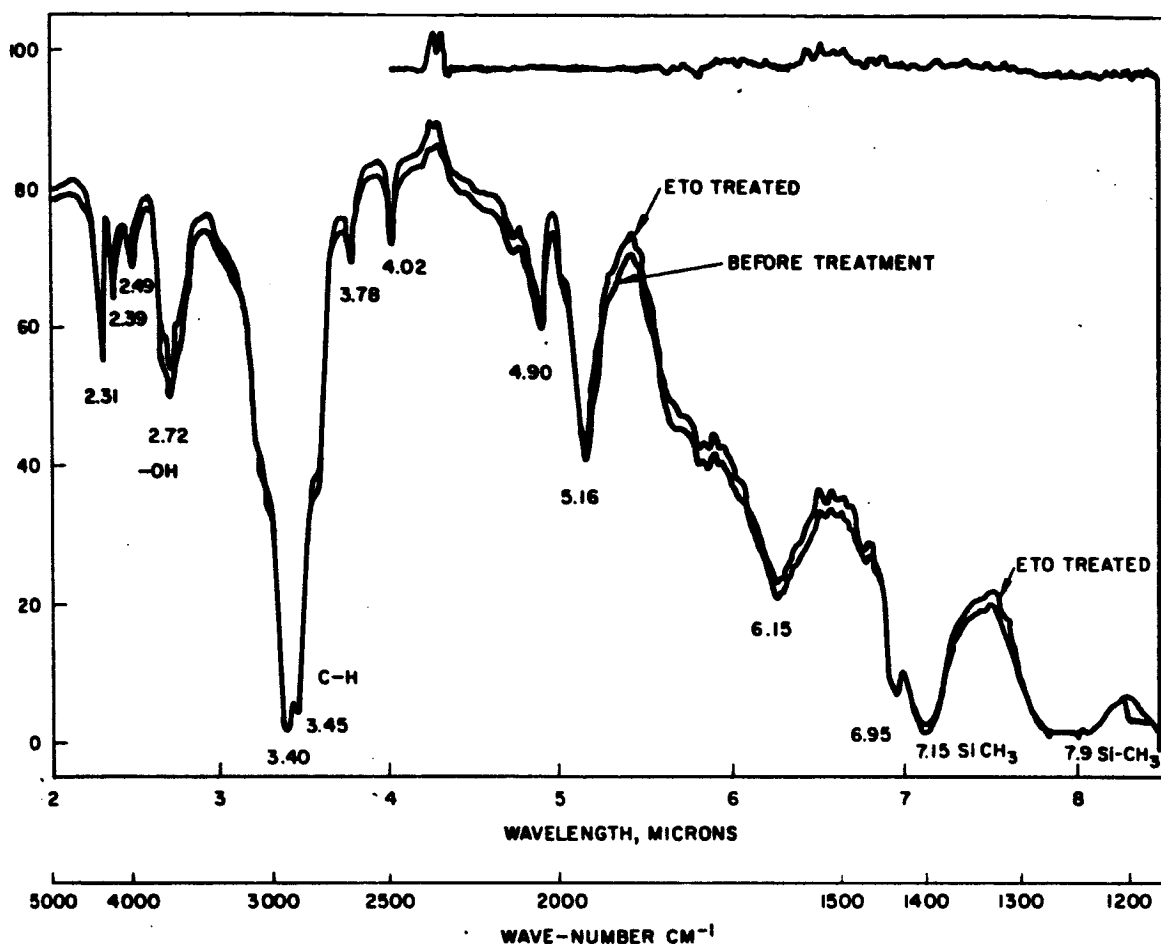


Figure 9. Infrared spectra of polydimethylsiloxane films before and after exposure to ethylene oxide.

Epoxy Polymers

Two pure epoxy polymers were prepared for study in the mechanism portion of the program.

Formulation 1

Epon 828 resin	100 parts
Methylnadic anhydride	75 parts

Formulation 2

Shell X-24 (diglycidyl ether of Bisphenol A)	25 parts
Metapheynlene diamine	8 parts

A sorption study was performed on formulation 1 using pure ethylene oxide gas. This study showed that while the epoxy material took up only 1.07 weight percent ethylene oxide, it retained 0.72 percent after vacuum exposure. A number of commercial products in the screening portion of the program showed a noticeable softening after gas exposure, indicating that a chemical gas-material interaction occurs to some extent. In general, subsequent heat cycling tends to restore the original hardness values.

Sorption studies on epoxy formulation 2 were performed with the 12 percent ETO-88 percent Freon 12 rather than with pure ethylene oxide. The original test plan called for exposing materials to both pure ETO and to ETO-Freon 12, however, program funding limitations did not allow this.

Formulation 2 adsorbed 0.026 weight percent sterilant gas and retained 0.014 percent after vacuum exposure. During vacuum exposure, the outgassed products were trapped in a liquid nitrogen trap. Infrared analysis showed that the composition of the outgassed products were essentially the same as the initial sterilant gas mixture.

Two commercial reinforced epoxy products were heat cycled before they were exposed to sterilant gas. Data in Tables 18 and 19 show that the final tensile strength of XP-206 was the same for both orders of exposure. Micarta H2497 showed a loss of 3.3 percent tensile when exposed to gas first, and a gain of 1.3 percent tensile when exposed to heat cycling first. This indicates that chemical interaction occurred to a small extent.

Phenolic Polymer

A pure phenolic polymer of the Resole type was synthesized for study; details of the synthesis are given in Appendix B. Sorption studies performed on the pure polymer using the 12 percent ETO-88 percent Freon 12 sterilant mixture, showed the material has a very low order of absorption, less than 0.001 percent by weight, and indicates the material is relatively inert to gas reaction.

Polyurethane

Sorption studies were conducted on a pure polyurethane polymer whose composition is given below:

Polyurethane Formulation

Dow P-400 (polyethylene glycol)	40 parts
Trimethylol propane	5 parts
2, 4 diisocyanotoluene	-----

Exposure to the sterilant gas mixture caused a 0.039 percent weight gain and subsequent vacuum treatment left a residue of 0.005 percent sterilant gas. Analysis of the outgassed products showed that the polymer had retained five mole percent ethylene oxide.

H-Film

A sample of DuPont H-Film, polyimide, was equilibrated with the sterilant gas mixture. Analysis of the residual gas above the H-Film showed that most of the ethylene oxide had been preferentially absorbed by the polymer. The material was outgassed by vacuum and an analysis of the outgassed products showed that the H-Film did not release the sorbed ethylene oxide. Data from the screening portion of the program indicates that H-Film undergoes a large change in mechanical properties during gas exposure (50 percent tensile loss). Subsequent heat cycling restores some of the original tensile strength (17 percent net loss after heat), thus indicating that heat is effective in desorbing ethylene oxide from the polymer.

Product and Manufacturer	Polymer Type	Control	After ETO-Freon 12 Exposure				After ETO-Freon 12 Exposure And Dry Heat Cycling				
		Hardness Shore A or D	Δ% Wt.	Δ% Vol.	Δ% Shear	Hard Shore	Δ% Wt.	Δ% Vol.	Δ% Shear	Hard Shore	
Ecobond 55/11 Emerson Cuming	Epoxy/Amine	84 D	-	-	-	-12.0	-	-	-	+ 8.9	-
Ecobond 57/C Emerson Cuming	Epoxy/Amine (Silver)	81 D	0.1	2.1	1.4	79 D	-0.56	1.00	- 2.5	85 D	
Epon 8/A Shell Chemical	Epoxy/Amine	86 D	0.34	-0.6	26.0	80 D	-0.2	-1.1	48.0	79 D	
Epon 828/A Shell Chemical	Epoxy/Amine	84 D	0.09	-	21.0	-	-0.1	-	- 1.7	82 D	
Epon 828/Z Shell Chemical	Epoxy/Amine	86 D	0.0	0.26	- 5.5	-	-0.26	-1.7	- 4.4	85 D	
Epon 901/B-1 Shell Chemical	Epoxy	84.5 D	0.69	-0.72	- 5.1	84	-0.48	-1.2	11.0	86 D	
Epon 901/B-3 Shell Chemical	Epoxy	72 D	0.31	0.83	-12.0	86 D	-0.28	-0.04	- 0.01	85 D	
E-Solder 3022 Epoxy Prod.	Epoxy/Silver	76 D	0.15	0.47	3.4	86 D	-1.2	-1.0	-15.0	86 D	
FM-96 Arco, Cyanamid	Epoxy-Nylon	83 D	1.4	1.37	-13.0	86 D	0.84	-0.65	-10.5	83 D	
RTV 108 General Elect.	Silicone	38 A	-0.28	0.82	-	37 A	-3.0	-2.9	+32	41 A	
RTV 140 General Elect.	Silicone	38 A	0.1	2.25	-	37 A	-0.20	-0.1	+29	40 A	
RTV 891 Dow-Corning	Silicone	38 A	0.08	1.26	-	38 A	-0.25	-0.02	-3.1	35 A	

Table 2. Change in properties of adhesives caused by gas exposure and dry heat cycling.

Product Name	Manufacturer	Type	Exposure	Cycles Per Milligram	% Change Cycles/Mg
Alkanex 9522 Varnish	General Electric	Alkyd Polyester	Control	115	-
			Gas	115	0
			Gas + Heat	97.2	-15
Electrofilm 4306 Lube-Lok	Electrofilm Corp.	Phenolic MoS ₂	Control	12	-
			Gas	18.5	+54
			Gas + Heat	18.6	+55
Pyre-ML Varnish RK 692	Du Pont	Polyimide	Control	11	-
			Gas	18.3	+66
			Gas + Heat	20	+82

Table 3. Change in properties of coatings caused by gas exposure and dry heat cycling.

Product and Manufacturer	Polymer Type	Control		After ETO-FREON 12 Exposure					After ETO-FREON 12 Exposure and Dry Heat Cycling				
		Volume Resistivity (Vol.Ω)	Hardness Shore A	% Δ Wt.	% Δ Vol.	Hardness Shore A	% Δ Tensile Strength	Vol.Ω	% Δ Wt.	% Δ Vol.	Hardness Shore A	% Δ Tensile Strength	Vol.Ω
AMS 3303/60 Hadbar Inc.	Silicone (HDBR 4000/60)	3.5×10^{13}	45	0.05	0.05	48	-0.01	7.1×10^{13}	-0.01	0.05	51	-3.0	1.5×10^{13}
HADBAR 1000/80 Hadbar Inc.	Silicone	7.4×10^{13}	87	0.44	1.10	87	+0.40	9.3×10^{13}	-0.25	0.07	81	-9.25	1.3×10^{14}
HADBAR 4000/80 Hadbar Inc.	Silicone	6.2×10^{13}	68	0.23	0.30	73	8.00	1.2×10^{14}	-0.01	0.02	75	-1.0	1.6×10^{14}
HADBAR 5000/80 Hadbar Inc.	Silicone	6.5×10^{13}	52	0.11	+1.10	53	0.98	3.0×10^{14}	-0.10	0.31	52	1.82	5.1×10^{13}
HADBAR 7000-80 Hadbar Inc.	Silicone	3.9×10^{13}	73	0.03	0.02	78	-10.0	-	0.01	0.27	78	-14.7	4.2×10^{13}
L-308-80 Parker Seal Co.	Fluorosilicone "O" Ring	"O" Ring	85	0.04	0.0	-	-9.6	-	-0.33	-0.25	81	-7.7	-
L-449-6/60 Parker Seal Co.	Fluorosilicone "O" Ring	"O" Ring	66	0.05	-0.15	-	+3.1	-	-0.17	-0.15	65	-11.0	-
LS 53/70 Products Research	Fluorosilicone	4.9×10^{13}	76	0.05	0.65	79	+1.37	7.0×10^{13}	-0.05	-0.35	82	-3.40	2.3×10^{14}
MIL-R-5847/50 Rubatex	Silicone	9.4×10^{13}	58	0.0	1.00	58	8.7	6.5×10^{13}	-0.05	4.5	58	-6.9	1.6×10^{14}
MIL-R-5847C/70 Rubatex	Silicone	7.2×10^{13}	63	0.04	0.60	65	-2.30	7.2×10^{13}	0.01	-2.29	60	-5.80	3.0×10^{14}
PMP 6100 Pacific Moulded Prod.	Silicone	6.7×10^{15}	51	0.98	-0.72	52	-7.40	-	-0.02	-4.12	52	-27.0	6.0×10^{15}
PR 1930-1/2 Products Research	Silicone	8.5×10^{12}	54	-0.01	-0.1	57	-	1.3×10^{13}	-1.10	-1.60	53	1.5	9.3×10^{12}

Table 4. Change in properties of elastomers caused by gas exposure and dry heat cycling.

Product and Manufacturer	Polymer Type	Control		After ETO-FREON 12 Exposure					After ETO-FREON 12 Exposure and Dry Heat Cycling				
		Volume Resistivity (Vol Ω)	Hardness Shore A	% Δ Wt.	% Δ Vol.	Hardness Shore A	% Δ Tensile Strength	Vol. Ω	% Δ Wt.	% Δ Vol.	Hardness Shore A	% Δ Tensile Strength	Vol. Ω
PR 1930-2 Products Research	Silicone	1.4×10^{13}	72	-0.01	-1.88	73	-	9.6×10^{12}	-0.68	-5.32	74	-	1.0×10^{13}
RTV 11 General Electric	Silicone (1)	3.00×10^{13}	57	-0.01	0.20	58	-	2.0×10^{13}	-1.01	-0.76	59	-9.3	5.5×10^{13}
RTV 60 Dow Corning	Silicone (1)	6.3×10^{13}	71	0.04	-0.46	71.5	-	5.08×10^{13}	-0.28	-0.87	73	13.1	4.9×10^{13}
RTV 560 General Electric	Silicone (1)	4.5×10^{13}	54	-0.24	3.50	59	-	5.1×10^{13}	-1.84	-2.50	61	4.2	2.4×10^{13}
RTV 615 A/B General Electric	Silicone	7.0×10^{13}	58	0.0	-1.70	53	-	6.68×10^{13}	-0.27	-1.75	64	-5.1	1.4×10^{15}
RTV 881 Dow Corning	Silicone (1)	6.8×10^{13}	52	0.02	0.0	56	-6.1	-	-0.24	-0.54	61	-8.4	8.7×10^{13}
S 417-7 Parker Seal	Silicone	"O" Ring	72	0.05	1.03	-	-1.14	-	0.09	-0.35	64	-3.5	-
SYLGARD 182 Dow Corning	Silicone (1)	6.0×10^{13}	64	0.04	0.10	65	-4.00	4.0×10^{12}	-0.5	-0.32	69	-52(2)	6.6×10^{13}
VITON B60 DuPont	Fluorocarbon	6.5×10^{13}	74	0.06	0.80	74	-12.0	4.9×10^{13}	-0.01	-0.15	77	-14.0	7.1×10^{14}
VITON B90 DuPont	Fluorocarbon	2.8×10^{15}	93	0.90	1.30	92	-15.0	6.6×10^{14}	0.20	0.45	93	+7.4	6.9×10^{14}

(1) Encapsulants

(2) Based on two test specimens, material has poor tear resistance - value probably not valid.

Table 4 (continued). Change in properties of elastomers caused by gas exposure and dry heat cycling.

Product Name and Manufacturer	Polymer Type	Control		After ETO-FREON 12 Exposure					After ETO-FREON 12 Exposure and Dry Heat Sterilization Cycling				
		Vol Ω	Shore A Hardness	% Δ Wt.	% Δ Vol	Shore A Hardness	% Δ Tensile Psi	Vol Ω	% Δ Wt.	% Δ Vol	Shore A Hardness	% Δ Tensile Psi	Vol Ω
RTV 11 General Electric	Silicone	3.0×10^{13}	57A	-0.01	0.20	58A		2.0×10^{13}	-1.01	-0.76	59	-20.3	5.5×10^{13}
RTV 60 General Electric	Silicone	6.3×10^{13}	71A	0.04	-0.46	71.5		5.1×10^{13}	-0.28	-0.87	73	-12.1	14.9×10^{13}
RTV 881 Dow Corning	Silicone	6.8×10^{13}	52	0.02	0.0	56	-6.1	6.1×10^{13}	-0.24	-0.54	61	-8.4	8.7×10^{13}
STYCAST 1095/11 Emerson Cuming	Epoxy/Amine	9.3×10^{14}	78	0.08	0.43	79	+11	8.1×10^{14}	-0.70	-5.3	76	+11.7	1.3×10^{15}
STYCAST 2651/11 Emerson Cuming	Epoxy/Amine	1.6×10^{15}	86	0.04	+0.51	87.5	+4.4 Shear	9.2×10^{14}	-0.16	-4.6	85	+14 Shear	2.3×10^{15}
SYLGARD 182 Dow Corning	Silicone	6.0×10^{13}	64	0.04	0.10	65	-4.0	4.0×10^{13}	-0.5	-0.32	69	-52 ⁽¹⁾	6.6×10^{13}

(1) Based on limited testing; material has poor tear resistance - value may not be valid.

Table 5. Changes in encapsulants produced by exposure to gas followed by dry heat cycling.

Product and Manufacturer	Polymer Type	Control		After ETO-FREON 12 Exposure				After ETO-FREON 12 Exposure and Dry Heat Cycling			
		% Ultim. Elong.	Vol. Ω	$\Delta\%$ Wt.	$\Delta\%$ Tensile	% Ultim. Elong.	Vol. Ω	$\Delta\%$ Wt.	$\Delta\%$ Tensile	% Ultim. Elong.	Vol. Ω
H-Film DuPont	Polyimide 0.002	60	1.9×10^{15}	1.0	-50	-	3.0×10^{17}	-0.47	-17	87	3.2×10^{17}
Mylar Type D DuPont	Polyester 3 mil	80	1.4×10^{16}	-0.80	-6.0	-	1.1×10^{17}	-2.0	-24	125	1.7×10^{17}
Mylar Type D DuPont	Polyester 5 mil	62	9.3×10^{16}	-0.45	-7.5	73	9.3×10^{16}	-0.6	-2.3	75	1.1×10^{17}
Mylar 65 HS DuPont	Polyester	-	7.4×10^{16}	+0.81	+72	-	8.3×10^{16}	-0.7	+82	-	1.1×10^{17}
Tedlar 200 Type 30B White DuPont .0020	Poly vinyl-fluoride	119	2.8×10^{15}	-0.30	-25	130	1.9×10^{15}	-0.6	-18	108	2.7×10^{16}

Table 6. Changes in properties of films caused by gas exposure and dry heat cycling.

Product and Manufacturer	Polymer Type and Alternate Designation	Control		After ETO-FREON 12 Exposure						After ETO-FREON 12 Exposure and Dry Heat Cycling							
		Hardness Shore D	Vol Ω	Δ% Wt.	Δ% Vol.	Hardness Shore D	Δ% Tensile	Δ% Flex.	Δ% Elast. Modulus	Vol Ω	Δ% Wt.	Δ% Vol.	Hardness Shore D	Δ% Tensile	Δ% Flex.	Δ% Elast. Modulus	Vol Ω
Diall FS-4 Mesa Plastics	Diallyl phthalate-Glass	91	4.9×10 ¹³	0.16	-1.4	89	-7.6	-3.9	-6.5	2.7×10 ¹³	-0.41	-1.2	90.5	-18	-13	-64	3.5×10 ¹³
Diall FS-10 Mesa Plastics	Diallyl phthalate-Glass	91	1.4×10 ¹⁵	0.07	-0.84	91	3.5	5	-4.3	3.56×10 ¹⁴	-0.56	-1.1	91	-17	-12	-63	2.0×10 ¹⁵
EG 758T Mica Corp.	Epoxy/Glass/Cu MIL-F-13949 FLOE	83	(1)	-0.05		83	-13.2	-2.1	-4.7	(1)	-0.34		85	1.1	-13.5	-30	(1)
FG-91 L/J Am. Reinforced Plast.	Phenolic/Glass	90	4.5×10 ¹³	0.01	-1.5	90	9.6	10.1	3.5	-	-1.9	-2.7	92	-7	-2.5	-0.5	7.6×10 ¹³
Laminite EG 752 Mica Corp	Epoxy/Glass	92	2.4×10 ¹³	-0.01	-1.1	92	-9.4			1.2×10 ¹⁴	0.0	-6.7	92	-14			9.2×10 ¹²
Lam. Grade H-5834 Westinghouse	Phenolic/Glass	93	4.0×10 ¹⁴	0.14	5.1	92.5	-3.3	3.4	-2.4	3.0×10 ¹⁴	0.15	3.7	93	-1.5	+6.5	-3.9	1.7×10 ¹⁴
Laminite NS Plastic Center	Phenolic/Nylon	86	1.9×10 ¹⁴	0.03	1.0	85.5	6.7	-9.3	9.6	7.18×10 ¹³	-3.2	-2.3	88	4.8	34.4	56	2.86×10 ¹⁴
Lam. 501J Budd Co.	Epoxy/Glass/Cu	89	9.5×10 ¹⁴	0.02	3.4	90	4.2	10.8	6.3	2.6×10 ¹⁵	-0.02	2.39	91	-2.1	14.6	1.7	1.4×10 ¹⁵
Micarta Grade 238 Westinghouse	Phenolic/Linen	91	3.5×10 ¹²	0.1	3.0	90	1.8	-5.5	-3.3	1.24×10 ¹²	-0.46	-0.7	91	6.5	-5.5	3.1	2.4×10 ¹²
Micarta GX Westinghouse	Epoxy/Glass H17480	90	6.1×10 ¹³	0.01	2.0	90	3.8	-5.2	-14	9.45×10 ¹³	-0.96	0.02	89	5.2	-1.5	-13.4	3.0×10 ¹⁴
Micarta H-2497 Westinghouse	Epoxy/Glass G-11	84.5	4.9×10 ¹⁵	-0.01			-1.3	-2.5	-2.3	6.9×10 ¹⁵	-0.2		84	-3.3	-16.2	-28	7.3×10 ¹⁵
Micarta J.E-221 Westinghouse	Phenolic/Linen	90	1.0×10 ¹⁴	-0.8	0.9	90	3.8		-3.0	1.1×10 ¹⁴	-3.0	-1.7	91	8.1	-9	+3.5	4.3×10 ¹⁵
Micarta I457 Westinghouse	Epoxy/Glass G-10	90	1.5×10 ¹⁵	0.2	2.1	91	2.2	4.0	5.3	9.1×10 ¹³	-0.38	3.5	91	2.22	-4.1	4.6	7.2×10 ¹⁴
XP-206 3M	Epoxy/Glass	34	4.7×10 ¹⁵	0.14		34	-1.76	3.5	7.4	4.7×10 ¹⁵	-0.18		34	5.1	-7.4	4	5.5×10 ¹⁵

(1) Copper clad. Metal particles embedded in polymer layer during sawing operation produced unreliable data.

Table 7. Change in properties of reinforced and unreinforced laminates caused by gas exposure and dry heat cycling.

Product and Manufacturer	Type	Exposure	Viscosity Centistokes	%Δ Weight	Dielectric Breakdown KV	%Δ KV	Rating
DC-5 Grease Dow Corning	Phenyl methyl-siloxane	Control	-	-	14.4	-	C
		Gas	-	+4.0	12.2	-15.2	
		Gas & Heat	-	+0.8	11.2	-22.2	
DC-200 350CS Dow Corning	Poly dimethyl-siloxane	Control	325.9	-	17.0	-	NC
		Gas	354.9	-0.03	17.4	+2.4	
		Gas & Heat	403.3	Nil	15.8	-7.1	
Versilube F-50 General Electric	Silicone	As Received	-	-	4.7	-	C
		Control ⁽¹⁾	78.8	-	18.1	-	
		Gas	63.0	Nil	19.1	+5.5	
		Gas & Heat	77.3	Nil	15.1	-16.6	

(1) D-treated with vacuum and heat to remove impurities.

Table 8. Change in properties of lubricants caused by gas exposure and dry heat cycling - product ratings.

Product	Manufacturer	Type	Rating	Criteria
Eccobond 55/11	Emerson Cuming	Epoxy/Amine	Insufficient Data	
Eccobond 57/C	Emerson Cuming	Epoxy/Amine Silver	C	Surface stains and blisters after gas exp.
Epon 8/A	Shell Chemical	Epoxy/Amine	C	
Epon 828/Z	Shell Chemical	Epoxy/Amine	C	
Epon 901/B-1	Shell Chemical	Epoxy	C	
Epon 901/B-3	Shell Chemical	Epoxy	C	
E-Solder 3022	Epoxy Prod	Epoxy/Silver	M	Weight loss = 1.2% Badly stained
FM-96	Am. Cyanamid	Epoxy/Nylon	C	
RTV 108	General Electric	Silicone	M	Weight loss = 3.0%
RTV 140	General Electric	Silicone	C	
RTV 891	Dow Corning	Silicone	C	

Table 9. Compatibility ratings - adhesives.

Product	Manufacturer	Type	Rating	Criteria - Comments
AMS 3303/60	Hadbar Inc.	Silicone	M	Hardness +6
Hadbar 1000/80	Hadbar Inc.	Silicone	M	Hardness -6 $\Delta\%$ Elongation - 29%
Hadbar 4000/80	Hadbar Inc.	Silicone	M	Hardness +7
Hadbar 5000/80	Hadbar Inc.	Silicone	C	
Hadbar 7000/80	Hadbar Inc.	Silicone	C	
L-308-80	Parker Seal Co.	Fluorosilicone	M	Elongation Loss - 26%
L-449-6/60	Parker Seal Co.	Fluorosilicone	C	
LS-53/70	Products Research	Fluorosilicone	M	Hardness +6
MIL-R -5847/50	Rubatex	Silicone	C	
MIL-R -5847C/70	Rubatex	Silicone	C	
PMP 6100	Pacific Moulded Prod.	Silicone	M	Tensile Loss - 27%
PR 1930-1/2	Products Research	Silicone	C	
PR 1930-2	Products Research	Silicone	C	
RTV 560	General Electric	Silicone	M	Hardness +7
RTV 615 A/B	General Electric	Silicone	M	Hardness +6
S 417-7	Parker Seal Co.	Silicone	NC	Elongation Loss - 60% Hardness Change - 8
VITON B60	DuPont	Fluorocarbon	M	Elongation Loss - 26%
VITON B90	DuPont	Fluorocarbon	C	

Table 10. Compatibility Ratings - Elastomers

Product	Manufacturer	Type	Rating	Criteria
RTV 11	General Electric	Silicone	M	Weight Loss - 1.01% (Tensile Loss - 20.3%)
RTV 60	General Electric	Silicone	C	
RTV 881	Dow Corning	Silicone	C	
StyCAST 1095/11	Emerson Cuming	Epoxy/Amine	M	Volume shrinkage = 5.3%
StyCAST 2651/11	Emerson Cuming	Epoxy/Amine	M	Volume Shrinkage = 4.6%
Sylgard 182	Dow Corning	Silicone	C*	* Tentative rating

Table 11. Compatibility ratings - Encapsulants.

Product	Manufacturer	Type	Rating	Criteria
H-Film	DuPont	Polyimide	C	Tensile Loss = 24% Weight Loss = 2%
Mylar Type D 3 mil	DuPont	Polyester	M	
Mylar Type D 5 mil	DuPont	Polyester	C	NOTE: H-Film and Tedlar showed appreciable losses in tensile proper- ties after gas exposure
Mylar 65 HC	DuPont	Polyester	C	
Tedlar 200 Type 30B White	DuPont	Poly vinyl- fluoride	C	

Table 12. Compatibility ratings - Films.

Product	Manufacturer	Type	Rating	Criteria
Diall FS-4	Mesa Plastics	Diallyl Phthalate	C	
Diall FS-10	Mesa Plastics	Diallyl Phthalate	C	
EG 758T	Mica Corp.	Epoxy/Glass Cu Clad	C	
FG-91 LD	Am. Reinforced Plas.	Phenolic /Glass	M	Weight Loss - 1. 9%
Laminate EG752	Mica Corp.	Epoxy /Glass	C	
Lam Grade H-5834	Westinghouse	Phenol/Glass	C	
Laminate NS	Plastic Center	Phenol/Nylon	M	Weight Loss - 3. 2%
Laminate 500J	Budd Co.	Epoxy /Glass Cu Clad	C	
Micarta Grade 238	Westinghouse	Phenol/Linen	C	
Micarta GX	Westinghouse	Epoxy /Glass	C	
Micarta H-2497	Westinghouse	Epoxy /Glass	C	
Micarta LE-221	Westinghouse	Phenol/Linen	M	Weight Loss - 3. 0%
Micarta 8457	Westinghouse	Epoxy /Glass	C	
XP-206	3M	Epoxy /Glass	C	

Table 13. Compatibility Ratings - Laminates

Product and Manufacturer	Type	Control		After Gas		After Gas & Heat		
		Hardness Shore D	% Δ Wt.	% Δ Vol.	% Δ Wt.	% Δ Vol.	Hardness Shore D	Hardness Shore D
Celcon Celanese Corp.	Polycarbonate	81	0.02	1.88	-0.58	-0.84	81	
GE PPO General Electric	Polyphenylene oxide	83	0.17	0.36	-0.01	-0.1	83	
Penton Hercules Powder	Chlorinated polyether	80	0.01	0.30	-0.57	-0.79	80	
Polysulfone Union Carbide	Polysulfone	82	0.04	0.06	-0.17	-0.42	84	
Teflon DuPont	Tetrafluoroethylene	60	0.01	0.0	-0.01	-0.04	60	

Table 14. Change in properties of miscellaneous JPL submitted products.

Material	Composition	ETO Taken Up Weight Percent	ETO Retained After Vacuum Exposure Weight Percent
Silicone formulation 1 (polydimethyl phenyl- methyl siloxane base)	C 1962-34 triethoxymethyl silane dibutyltin dilaurate 100 parts 8 parts 2 parts	3.70	0.40
Silicone formulation 2 (polydimethyl silox- ane base)	C 1090-61A triethoxymethyl silane dibutyltin dilaurate 100 parts 8 parts 3 parts	8.30	0.00
Silicone formulation 3 (polydimethyl silox- ane base)	C 1090-60Z triethoxymethyl silane dibutyltin dilaurate 100 parts 8 parts 2 parts	3.00	0.04
Silicone formulation 4 (polydimethyl silox- ane base)	C 1090-60A triethoxymethyl silane dibutyltin dilaurate powdered silica 100 parts 8 parts 2 parts 50 parts	2.61	0.06

Table 15. ETO sorption by pure silicone polymers.

Material	Control			After Gas			After Gas and Heat		
	Tensile psi	% Ult. Elong.	Shore A Hard.	Tensile psi	% Ult. Elong.	Shore A Hard.	Tensile psi	% Ult. Elong.	Shore A Hard.
AMS 3303/60	1030	658	45	1017	620	48	1000	570	51
HDBR 4000/80	913	300	68	1008	195	73	922	283	75
HDBR 7000/80	932	208	73	835	175	78	795	200	78

Table 16. Properties of silicone products exposed to sterilant gas followed by dry heat cycling.

Material	After Heat Cycle			After Heat and Gas		
	Tensile psi	% Ult. Elong.	Shore A Hard.	Tensile psi	% Ult. Elong.	Shore A Hard.
AMS 3303/60	1065	641	53	1090	563	55
HDBR 4000/80	920	300	75	880	300	77
HDBR 7000/80	938	190	79	900	200	80.5

Table 17. Properties of silicone products exposed to dry heat cycling first and then to sterilant gas.

Material	Control Tensile psi x 10 ³	After Gas Exposure Tensile psi x 10 ³	After Gas and Heat Tensile psi x 10 ³
Micarta H-2497 Epoxy-fiberglass	45.3	44.7	43.8
XP-206 Epoxy-fiberglass	45.2	46.0	47.5

Table 18. Properties of epoxy products exposed to sterilant gas followed by dry heat cycling.

Material	After Heat Cycle Tensile psi x 10 ³	After Heat Cycle and Gas Tensile psi x 10 ³
Micarta H-2497 Epoxy-fiberglass	48.2	45.9
XP-206 Epoxy-fiberglass	43.1	47.5

Table 19. Properties of epoxy products exposed to dry heat cycling first and then to sterilant gas.

APPENDICES

APPENDIX A	Preparation, Application and Cure of Polymeric Product Test Specimens
APPENDIX B	Pure Polymer Synthesis and Formulation
APPENDIX C	Test Data

APPENDIX A

PREPARATION, APPLICATION AND CURE OF POLYMERIC PRODUCT TEST SPECIMENS

This appendix describes in detail the techniques used to fabricate the test specimens used in this program. Materials are divided into functional categories, and applicable information on curing agent concentrations, methods of application or casting, cure conditions, and substrate preparation given for each material. Products which arrived from the manufacturer in finished form are not covered in this appendix. All materials were mixed, applied, and cured according to the manufacturer's recommended instructions. In casting operations, the use of organic parting compounds was held to a minimum; and teflon molds were used for the majority of the products. Materials to be cast into sheet form were degassed immediately after mixing, and again after they had been cast, just prior to cure.

I. ADHESIVES

A. Mechanical Testing

1. Substrate Preparation

6061T4 aluminum coupons, 1" x 4" x 0.063", were solvent wiped, and etched in an aqueous solution of 2.5 percent sodium dichromate, 28 percent sulfuric acid at 145°F for 10 minutes. The coupons were flushed with tap water, rinsed with deionized water, and air dried.

2. Adhesive Application and Cure

Adhesive products were mixed (see table for ratios) and uniformly spread over the end 0.5 inch of the aluminum coupons. The coupons were assembled, clamped into a holding fixture under two pounds pressure and cured. Curing agent concentrations and cure conditions are given below.

Product and Manufacturer	Type	Curing Agent Conc. Percent by Weight	Cure Temperature and Time
Eccobond 55 (Emerson Cuming)	Epoxy - Amine	17% No. 11	300°F/0.5 Hrs
Eccobond 57C (Emerson Cuming)	Epoxy - Silver - Amine	50% A + 50% B	225°F/0.5 Hrs
Epon 8A (Shell)	Epoxy - Amine	5.6% A	200°F/1.5 Hrs
Epon 828A (Shell)	Epoxy - Amine	7.4% A	250°F/3 Hrs
Epon 828Z (Shell)	Epoxy - Amine	16.7% Z	300°F/4 Hrs
Epon 901/B-1 (Shell)	Epoxy	18.7% B-1	200°F/1 Hr
Epon 901/B-3 (Shell)	Epoxy	10% B-3	350°F/2 Hrs
E-Solder 3022 (Epoxy Products)	Epoxy - Silver	7.4% No. 18	200°F/1.5 Hrs.
FM-96 (Am. Cyanamid)	Epoxy - Nylon	Structural adhesive	350°F/1.5 Hrs ⁽¹⁾
RTV 108 (G. E.)	Silicone	Air cure	150°F/4 Hrs at 90% relative humidity followed by 24 hours at room temperature
RTV 140 (G. E.)	Silicone	Air cure	
RTV 891 (Dow Corning)	Silicone	Air cure	

(1) FM-96 specimens were cured under 10 pounds pressure.

B. Physical and Electrical Property Test Specimens

All adhesive materials were cast into discs, 1.5 inches in diameter, and 1/16 inch thick, using teflon lined molds. RTV 108, 140 and 891 are high viscosity pastes which must be exposed to atmospheric moisture to effect cure. The following technique was used to cast smooth surfaced, bubble free specimens.

1. The RTV material was carefully injected from the tube into the mold, keeping air entrapment to a minimum.
2. The mold top (polished aluminum plate) was covered with a wetted square of tissue paper and then placed, paper side down, on the filled mold. Pressure was exerted with clamps until the excess adhesive was extruded from between the plates; the clamped mold was then placed in a container of water for twenty minutes. Water, wicked up by the paper, served to cure the layer of adhesive in contact with it. The mold top was removed and the adhesive in the mold allowed the air cure for a minimum of 12 hours.

II. ELASTOMERS

Elastomeric materials, arriving in the uncured state were fabricated into sheet to facilitate testing. Curing agent concentrations and cure conditions are given below.

Product and Manufacturer	Type	Curing Agent Conc. Percent by Weight	Cure Temperature and Time
PR 1930-1/2 (Products Research)	Silicone	9% Accelerator	Room temp/4 hrs. 175°F/2 hrs.
PR 1930-2 (Products Research)	Silicone	9% Accelerator	Room temp/4 hrs. 175°F/2 hrs.
RTV 560 (G. E.)	Silicone	0.5% Thermolite 12	Room temp/4 hrs. 200°F/2 hrs.
RTV 615 (G. E.)	Silicone	91% A + 9% B	Room temp/4 hrs. 200°F/2 hrs.

III. ENCAPSULANTS

Physical, mechanical, and electrical test specimens were cut from sheets of encapsulant materials which had been cast in teflon lined molds. Stycast 2651/11 could not be fabricated into suitable sheet, and lap shear tests were performed in lieu of tensile tests. Discs were cast, as described in section IB Adhesives, for physical and electrical testing.

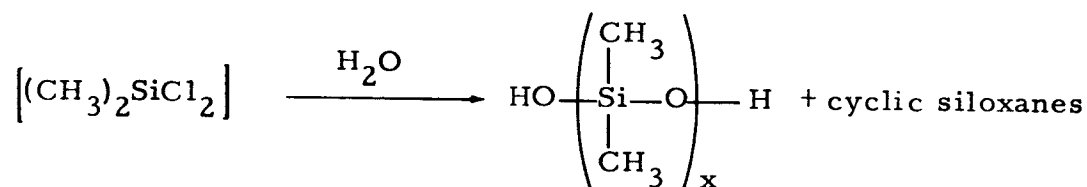
Product and Manufacturer	Type	Curing Agent Conc. Percent by Weight	Cure Temperature and Time
RTV 11 (G. E.)	Silicone	0.5% Ther- molite 12	Room temp/24 hrs. 175°F/24 hrs.
RTV 60 (G. E.)	Silicone	0.5% Ther- molite 12	Room temp/24 hrs. 175°F/2 hrs.
RTV 881 (Dow Corning)	Silicone	3.7% Catalyst	Room temp/4 hrs. 175°F/2 hrs
Stycast 1095/11 (Emerson Cuming)	Epoxy	20% No. 11 Hardner	150°F/18 hrs. 250°F/2 hrs.
Stycast 2651/11 (Emerson Cuming)	Epoxy	8% No. 11 Hardner	150°F/18 hrs. 220°F/3 hrs.
Sylgard 182 (Dow Corning)	Silicone	9% Curing Agent	Room temp/24 hrs. 175°F/2 hrs.

APPENDIX B

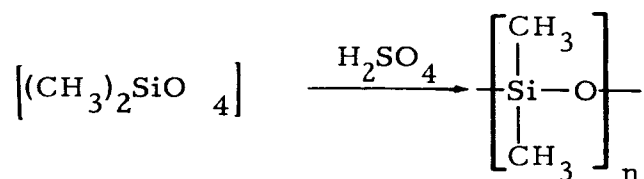
PURE POLYMER SYNTHESIS AND FORMULATION

I. SILICONE RESINS

The basic synthesis of the polydimethylsiloxane polymer can be represented by the reaction



The resulting polymer is a colorless viscous liquid which can easily be cured into an elastomeric material. Polydimethylsiloxanes can also be prepared by equilibration of cyclic dimethylsiloxanes with either acid or basic catalyst. When an acid catalyst such as sulfuric acid is used, the product has a lower viscosity than when a basic catalyst is employed. A typical equilibration reaction involves the reaction of octamethylcyclotetrasiloxane with concentrated sulfuric acid. This reaction can be written as follows:

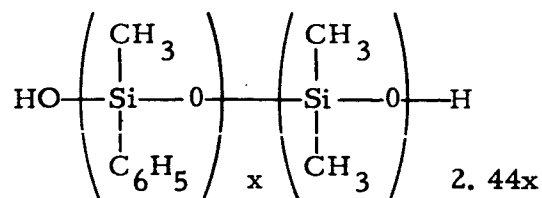


Polydimethylsiloxane C1090-60-A. - A six liter three-necked flask fitted with a stirrer, dropping funnel, and thermometer was filled with 3000 ml of water. One liter of dimethyldichlorosilane was added dropwise while stirring vigorously. The temperature was kept in the range of 15° - 25°C by cooling with an external ice-bath. After the addition the mixture was allowed to stand overnight. The upper layer was removed, washed and centrifuged to separate any occluded water.

The product was subjected to a distillation at atmospheric pressure. When the pot temperature reached 200°C vacuum was applied. The distillation was discontinued when the pot temperature reached 225°C under less than 1 mm pressure. The distillate which consisted mostly of cyclic siloxanes weighed 316 grams. The residue, a colorless heavy oil, weighed 225 grams and was the desired product.

Polydimethylsiloxane C1090-61A. - Into a 500 ml Erlenmeyer flask fitted with a magnetic stirrer were added 140 ml of octamethylcyclotetra-siloxane (b. p. 173° - 176°C/760 mm Hg), 24.9 ml of concentrated sulfuric acid and 70 ml of diethyl ether. The mixture was stirred for one day during which time considerable increase in viscosity was noted. The reaction product was washed with water a number of times to remove the acid. The ether solution was dried over anhydrous potassium carbonate and subjected to a distillation under vacuum. The distillation was discontinued when the pot temperature reached 210°C and a pressure of 1 mm Hg.

Polydimethyl phenylmethylsiloxane C1962-34. - A mixture consisting of dimethyldichlorosilane (435 ml) and phenylmethyldichlorosilane (180 ml) was added dropwise to 3000 ml of water while stirring vigorously. The temperature during the addition was kept below 35°C using an ice-bath. After the addition the mixture was stirred overnight. The siloxane was separated, washed with water until the washings were free of acid and then subjected to a vacuum distillation to remove more volatile siloxane fractions. The volatile constituents were chiefly cyclic siloxanes and weighed 122 g. When the pot temperature reached 225°C the distillation was discontinued. The residue weighed 236 g. Based on stoichiometry the structure of the polymer can be approximated by the following formula



Pure Silicone Polymer Formulations

Silicone Formulation 1

Polydimethyl phenylmethylsiloxane (C-1962-34)	100 parts
triethoxymethylsilane	8 parts
dibutyltin dilaurate	2 parts

The mixture was maintained at 50°C for 24 hours, then heated slowly under vacuum up to 150°C. It was kept at 150°C for 24 hours under vacuum to remove volatile constituents.

Silicone Formulation 2

polydimethylsiloxane (C1090-61A)	100 parts
triethoxymethylsilane	8 parts
dibutyltin dilaurate	3 parts

The mixture was kept at 50°C for 24 hours, then heated slowly under vacuum to 150°C and maintained at temperature for 24 hours under vacuum to remove volatile constituents.

Silicone Formulation 3

polydimethylsiloxane (C1090-60A)	100 parts
triethoxymethylsilane	8 parts
dibutyltin dilaurate	2 parts

The mixture was kept at 50°C, heated up slowly to 150°C and kept at that temperature for 48 hours under vacuum.

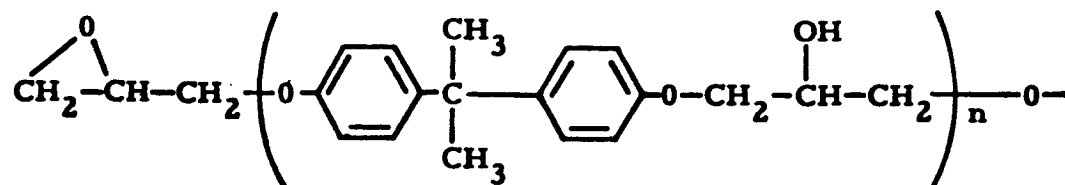
Silicone Formulation 4

polydimethylsiloxane (C1090-60A)	100 parts
triethoxymethylsilane	8 parts
dibutyltin dilaurate	2 parts
powdered silica, 270 mesh	50 parts

The mixture was cured for several days at room temperature and then heated up to 150°C and kept at this temperature for 72 hours under vacuum.

II. EPOXY RESIN

Many of the epoxy resins are based upon condensation polymers of epichlorohydrin and bisphenol A. These resins are characterized by the formula:



where the value of n determines the viscosity of the resin and other physical properties. There are over a hundred compounds available commercially that can cure these resins because of the presence of both epoxy groups and hydroxy groups as curing sites. For this study the Epon resin types which are marketed by Shell Chemical Corp. and have the above general formula were used. Epon 828 was chosen as the representative resin. It has an average molecular weight of 350, epoxy value of 0.5 and a hydroxyl value of 0.1. Methylnadic anhydride was chosen as a characteristic acid anhydride curing agent. It is a liquid at room temperature and can be readily mixed with the resin to form a more uniform material. Shell X-24 cured with metaphenylene diamine was chosen as a typical amine cured polymer.

Pure Epoxy Polymer Formulations

Epoxy Formulation 1

Epon 828 resin	100 parts
methylnadic anhydride	75 parts

The resin mixture was cured by heating to 150°C for 48 hours in air and then under vacuum at 100°C for 24 hours.

Epoxy Formulation 2

Shell X-24 (diglycidyl ether of Bisphenol A)	100 parts
metaphenylene diamine	32 parts

The two compounds were mixed thoroughly, warmed to effect solution, and placed in a mold. The specimen was cured by heating for 24 hours

at 165°F, then at 212°F for two hours, and finally at 250°F for 24 hours.

III. PHENOLIC RESIN

The following procedure was used to produce a pure phenolic resin of the Resole type. Into a 500 ml resin flask fitted with a thermometer, magnestirrer, Glas-col heater and condenser were added 94 g. of phenol, 132 g. of aqueous formaldehyde -37% by weight, and 4.7 g. of barium hydroxide octahydrate. The reaction mixture was heated at 70°C for several hours then allowed to stand at room temperature for 16 hours. It was brought to a pH of 6-7 with 10% sulfuric acid and subjected to a distillation at 60-70°C using a rotafilm evaporator. The distillation was discontinued when a drop placed on a hot plate surface readily formed a rubbery solid. The resin was molded into 3/16 inch slabs and cured by first heating at 165°F for 20 hours, then at 185°F for 24 hours followed by 24 hours at 212°F. It was removed from the mold and heated for an additional 24 hours at 250°F. The result was a hard slab of phenolic resin.

IV. POLYURETHANE RESIN

The pure polyurethane resin was prepared by mixing dry polyethylene glycol, P-400 Dow Chemical, 40 g., trimethylolpropane 5 g. and 2, 4-diisocyanotoluene. The mixture was placed in a 3/16 inch thick mold and cured overnight at room temperature, then at 168°F for 24 hours and finally for 24 hours at 250°F.

APPENDIX C

TEST DATA

The tables in this appendix present mechanical and electrical test data for commercial polymeric products screened in this program. The individual values are listed as well as the average value for each series. The average value appearing at the bottom of each column of mechanical test data is not necessarily an arithmetic average of all the values appearing above it; some values deviated markedly and were not used. The average values shown in this appendix were used to calculate the property changes given in the tables in the body of the report.

ADHESIVES

ECCOBOND 55/11, EMERSON AND CUMING, EPOXY

	Shear Strength psi x 10 ³	Hardness Shore D	Volume Resistivity and Thickness-in
Control	3.38 3.40 3.37 av. 3.38	84 84 84 84 85 83 84 83 86 av. 84 83	1.1 x 10 ¹⁵ ----- 0.075 Hartshorn sample holder
After ETO - Freon 12 exposure	2.93 2.75 2.90 2.78 av. 2.84		1.3 x 10 ¹⁵ ----- 0.077
After ETO - Freon 12 exposure and dry heat cycling	3.75 3.64 av. 3.69		9.7 x 10 ¹⁴ ----- 0.074

ECCOBOND 57 C, EMERSON AND CUMING, EPOXY

	Shear Strength psi x 10 ³	Hardness Shore D
Control	0.84 0.90 0.97 av. 0.92	81 80 80 81 82 av. 81
After ETO - Freon 12 exposure	0.92 0.94 0.93 av. 0.93	79 av. 79 79 78 80
After ETO - Freon 12 exposure and dry heat cycling	0.88 0.80 0.96 av. 0.89	85 85 85 av. 85 86

EPON 8/A, SHELL CHEMICAL, EPOXY

	Shear Strength psi x 10 ³	Hardness Shore D
Control	2.25 2.24 2.29 av. 2.26	86 87 86 86 83 av. 86 82
After ETO - Freon 12 exposure	2.75 2.97 av. 2.86	82 79 79 av. 80 80
After ETO - Freon 12 exposure and dry heat cycling	3.35 3.62 3.50 av. 3.48	76 81 75 av. 79

ADHESIVES

EPON 828/A, SHELL CHEMICAL, EPOXY

	Shear Strength psi x 10 ³	Hardness Shore D
Control	2.98 3.10 2.87 av. 2.98	84 84 84 85 83 av. 84
After ETO - Freon 12 exposure	1.87 3.75 av. 3.75	No measurements taken
After ETO - Freon 12 exposure and dry heat cycling	2.96 2.61 3.20 av. 2.93	80 84 82 82 82 81 83 av. 82

EPON 828/Z, SHELL CHEMICAL, EPOXY

	Tensile and Flexural Strength psi x 10 ³		Shear Strength x 10 ³	Elastic Mod. x 10 ⁵	Hardness Shore D
	T	F			
Control	8.95 9.36 9.15 9.20 av. 9.16	25.5 28.3 26.3 av. 27.0	2.92 3.29 av. 3.10	4.4 4.45 4.15 av. 4.3	86 86 86 87 87 av. 86
After ETO - Freon 12 exposure	8.97 8.43 8.70 av. 8.69		2.65 3.22 av. 2.93		No measurements taken
After ETO - Freon 12 exposure and dry heat cycling	8.90 9.10 8.40 av. 8.76		2.94 2.92 2.99 av. 2.95		85 86 85 85 86 av. 85 85

EPON 901/B-1, SHELL CHEMICAL, EPOXY

	Shear and Flexural Strength psi		Hardness Shore D	Volume Resistivity and Thickness-in
	S x 10 ⁵	F x 10 ³		
Control	3.15 3.13 3.14 av. 3.14	9.55 9.31 av. 9.40	84 84 84 85 84 av. 85 84.5	1.0 x 10 ¹⁴ ----- 0.068 Hartshorn sample holder
After ETO - Freon 12 exposure	2.76 3.20 av. 2.98	8.72 8.86 av. 8.79	84 84 84 84 85 85 av. 84	3.2 x 10 ¹⁴ ----- 0.068
After ETO - Freon 12 exposure and dry heat cycling	3.49 3.66 av. 3.58	10.2 9.36 11.20 av. 10.25	87 86 88 84 86 av. 86	1.0 x 10 ¹⁴ ----- 0.069

ADHESIVES

EPON 901/B-3, SHELL CHEMICAL EPOXY

	Shear Strength psi x 10 ³	Hardness Shore D	Volume Resistivity and Thickness-in
Control	3.09 3.05 3.06 av. 3.07	71 73 72 74 71 71 av. 72	1.8 x 10 ¹⁴ ----- 0.067 Hartshorn sample holder
After ETO - Freon 12 exposure	2.74 2.66 av. 2.70	86 86 85 83 85 86 av. 86	2.4 x 10 ¹⁴ ----- 0.067
After ETO - Freon 12 exposure and dry heat cycling	2.85 3.20 3.16 av. 3.07	86 86 86 87 87 86 av. 85	9.7 x 10 ¹³ ----- 0.068

EPOXY SOLDER 3022, EPOXY PRODUCTS COMPANY, EPOXY

	Shear Strength psi x 10 ³	Hardness Shore D
Control	2.00 2.13 2.05 av. 2.06	73 88 76 77 av. 76
After ETO - Freon 12 exposure	2.04 2.23 av. 2.13	86 87 85 av. 86
After ETO - Freon 12 exposure and dry heat cycling	1.58 1.69 av. 1.63	85 86 86 av. 86

FM 96, BLOOMINGDALE RUBBER, EPOXY

	Shear Strength psi x 10 ³	Hardness Shore D
Control	2.80 3.34 3.64 av. 3.21	81 84 78 83 av. 83
After ETO - Freon 12 exposure	3.34 3.24 av. 3.29	86 86 80 av. 86
After ETO - Freon 12 exposure and dry heat cycling	2.43 3.96 3.82 av. 3.80	80 86 83 av. 83

ADHESIVES

RTV 108, GENERAL ELECTRIC, SILICONE

	Tensile Strength psi	Hardness Shore A	Volume Resistivity and Thickness-in
Control	218	40	1.1×10^{15}
	220	38	-----
	280	39	0.081
	av. 242	38 av. 38	Hartshorn sample holder
After ETO - Freon 12 exposure	211	38	6.7×10^{14}
	192	36	-----
	207	38	0.081
	av. 203	av. 37	
After ETO - Freon 12 exposure and dry heat cycling	311	42	6.8×10^{14}
	301	41	-----
	348	41	0.079
	av. 320	av. 41	

RTV 140, DOW CORNING, SILICONE

	Shear	Hardness Shore	Volume Resistivity and Thickness-in
Control	337	38 37	3.2×10^{14}
	295	39 37	-----
	313	38.5 37	0.071
	av. 315	38	0.073
		38 av. 38	Hartshorn sample holder
After ETO - Freon 12 exposure		39 36	2.8×10^{14}
		38 av. 37	-----
		35	0.072
			0.073
After ETO - Freon 12 exposure and dry heat cycling	438	40 41	3.0×10^{14}
	428	39 40	-----
	306	40	0.072
	405	39 av. 40	0.072
	av. 400		

RTV 891, DOW CORNING, SILICONE

	Shear Strength psi	Hardness Shore A	Volume Resistivity and Thickness-in
Control	350	37 37.5	6.0×10^{14}
	338	38 38	-----
	350	37 38	0.078
	484	32	0.076
	av. 347	39 av. 38	Hartshorn sample holder
After ETO - Freon 12 exposure		36 38	7.3×10^{14}
		36 37	-----
		37	0.079
		38 av. 38	0.073
After ETO - Freon 12 exposure and dry heat cycling	310	30 38	3.5×10^{14}
	369	33 37	-----
	269	38	0.079
	400	35 av. 35	0.075
	av. 336		

ELASTOMERS

AMS 3303/60, HADBAR 4000/60, SILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in
Control	875	550	47 46.5	3.5×10^{13}
	1040	625	46 41.5	
	1050	675	45	0.068 0.066
	1160	675	45 av. 45	0.064 0.064
	av. 1030	av. 630		0.062 av. 0.065
After ETO - Freon 12 exposure	1020	650	48 47.5	7.1×10^{13}
	980	550	49 51.5	0.061 0.064
	1190	650	48 av. 48	0.064 0.062
	1050	625		0.063 av. 0.067
	av. 1017	av. 620		
After ETO - Freon 12 exposure and dry heat cycling	885	525	53 53	1.5×10^{13}
	975	725	50 53	
	943	575	54	0.069 0.069
	1000	600	50 av. 51	0.070 0.068
	av. 1000	av. 570		0.070 av. 0.068

COMPOUND 1000-80, HADBAR, SILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in
Control	899	175	85 86	7.4×10^{13}
	1120	250	88 87	
	988	200	87 av. 87	0.061 0.061
	988	250		0.061 0.061
	av. 965	av. 200		0.062 av. 0.061
After ETO - Freon 12 exposure	1000	150	86 86	9.3×10^{13}
	1000	160	87 88	
	1050	160	86	0.061 0.060
	1020	160	88 av. 87	0.061 0.062
	av. 998	av. 158		0.061 av. 0.061
After ETO - Freon 12 exposure and dry heat cycling	942	135	80 80	1.3×10^{14}
	794	105	80 81	
	934	157	82	0.062 0.060
	1015	130	82 av. 81	0.060 0.061
	av. 876	av. 141		0.060 av. 0.061

COMPOUND 4000-80, HADBAR, SILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in
Control	935	300	67 68	6.2×10^{13}
	890	275	68 67.5	
	915	300	69	0.071 0.072
	880	300	67.5 av. 68	0.072 0.072
	av. 905	av. 295		0.072 av. 0.072
After ETO - Freon 12 exposure	995	180	72 73.5	1.2×10^{14}
	1010	200	73 73	
	995	200	74.5	0.071 0.069
	1020	190	74 av. 73	0.070 0.068
	av. 1008	av. 195		0.069 av. 0.069
After ETO - Freon 12 exposure and dry heat cycling	765	250	77 76	1.6×10^{14}
	955	225	76 75	
	890	300	76 av. 75	0.070 0.068
	920	300		0.070 0.066
	av. 922	av. 283		0.068 av. 0.068

ELASTOMERS

COMPOUND 5000-50, HADBAR, SILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in
Control	850	475	51 53	6.5×10^{13}
	815	625	52	
	775	725	51 av. 52	0.071 0.065
	905	600		0.072 0.069
	av. 815	725		0.071 av. 0.069
After ETO - Freon 12 exposure	850	500	63 54	3.0×10^{14}
	840	525	62 53	
	820	450	52 53	0.065 0.072
	804	av. 492	51 av. 53	0.066 0.068
	av. 827			av. 0.068
After ETO - Freon 12 exposure and dry heat cycling	800	490	54 54	5.1×10^{13}
	902	525	54 52	
	852	490	52	0.070 0.071
	861	515	53 av. 52	0.072 0.066
	773	455		av. 0.070
	av. 838	av. 495		

COMPOUND 7000-80, HADBAR, SILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in
Control	940	200	74.5 74	3.9×10^{13}
	935	200	73 73	
	926	225	74 73	0.090 0.084
	av. 934	150	74 av. 73	0.089 0.086
		av. 208		0.086 av. 0.087
After ETO - Freon 12 exposure	740	175	78 78	2.1×10^{13}
	890	175	78 78	
	880		79 av. 78	0.076 0.077
	805	av. 175		0.076 0.078
	820			0.073 av. 0.076
	av. 835			
After ETO - Freon 12 exposure and dry heat cycling	768	200	83 80	4.2×10^{13}
	795	175	82 81	
	820	225	82 80	0.081 0.079
	899	av. 200	81 av. 78	0.085 0.081
	av. 795			0.085 av. 0.082

L-308-8, PARKER SEAL COMPANY, FLUOROSILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in
Control	970	390	85	"O" Ring
	900	350	86	
	890	380	84	
	av. 926	av. 373	av. 85	
After ETO - Freon 12 exposure	628	270		
	845	320		
	940	380		
	av. 804	av. 320		
After ETO - Freon 12 exposure and dry heat cycling	755	242	81	
	855	255	81	
	855	294	81	
	av. 821	av. 275	av. 81	

ELASTOMERS

L-449-6/60, PARKER SEAL COMPANY, FLUOROSILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in
Control	741 906 902 707 923 av. 836	141.5 134.0 154.5 136.5 av. 136	67 65 66 av. 66	"O" Ring
After ETO - Freon 12 exposure	965 830 725 860 930 av. 862	144 129 106 130 138 av. 129.4		
After ETO - Freon 12 exposure and dry heat cycling	789 658 789 av. 745	667 642 718 av. 680	63 66 67 av. 65	

LS-53/70, PLASTIC AND RUBBER PRODUCTS, FLUOROSILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in
Control	1230 1240 1180 1240 av. 1240	300 325 300 350 av. 308	76 73 78 78 74 av. 76	4.9×10^{13} ----- 0.064 0.063 0.067 0.066 av. 0.065
After ETO - Freon 12 exposure	1275 1236 1260 1190 av. 1257	325 325 325 325 av. 325	77 80 79 77 80 79 av. 79	7.0×10^{13} ----- 0.073 0.069 0.070 0.069 av. 0.070
After ETO - Freon 12 exposure and dry heat cycling	1170 1210 1200 1080 780 av. 1193	300 300 300 275 250 av. 300	83 83 83 81 82 82 80 83 av. 82	2.3×10^{14} ----- 0.068 0.070 0.068 0.072 av. 0.069

MIL-R-5847, CLASS II, GRADE 50, RUBATEX, SILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in
Control	813 600 738 825 860 av. 832	600 550 625 675 av. 612	63 63 62 63 63 av. 63	9.4×10^{13} ----- 0.063 0.063 0.063 0.064 0.062 av. 0.063
After ETO - Freon 12 exposure	863 794 879 840 896 834 av. 813	550 385 585 545 580 585 585 420 560 370	64 66 65 68 65 64 av. 65	6.5×10^{13} ----- 0.066 0.064 0.065 0.064 0.066 av. 0.065
After ETO - Freon 12 exposure and dry heat cycling	847 636 888 881 735 804 833 639 av. 783	530 530 555 495 440 av. 511	56 61 55 62 57 56 av. 60	1.6×10^{14} ----- 0.06 0.0620 0.06 0.0639 0.0631 av. 0.0629

ELASTOMERS

MIL-R-5847C, CLASS II/70, RUBATEX, SILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in
Control	855	600	58 58	7.2×10^{13}
	775	560	58	
	825	580	59 av. 58	0.064 0.063
	770	550	58	0.064 0.062
	av. 806	625 av. 583		av. 0.063
After ETO - Freon 12 exposure	835	570	58 59	7.2×10^{13}
	872	610	59	
	940	625	58 av. 58	0.063 0.063
	920	600	59	0.065 0.062
	815	610	58	av. 0.063
After ETO - Freon 12 exposure and dry heat cycling	752	550	57 58	3.0×10^{14}
	733	505	58 57	
	717	505	57	0.061 0.062
	754	505	58 av. 58	0.062 0.061
	796	565		0.063 av. 0.062

PMP 6100 (AMS 3301-40), PACIFIC MOULDED PROD., EPOXY

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in
Control	1170	500	51 51	6.7×10^{15}
	1140	440	51 51	
	1180	490	50	0.062 0.064
	1140	550	50 av. 51	0.062 0.064
	1160	530		av. 0.063
After ETO - Freon 12 exposure	av. 1150	500		
		av. 482		
	1110	520	51 52	
	995	480	52 53	
	1040	490	53	
After ETO - Freon 12 exposure and dry heat cycling	1110	530	53 av. 52	
	1070	510		
	av. 1065	506		
	767	380	52 50	6.0×10^{15}
	840	415	53 53	
	802	395	51	0.063 0.061
	925	425	54 av. 52	0.061 0.067
	868	415		0.065 av. 0.063
	av. 840	406		

PR-1930-1/2, PRODUCTS RESEARCH, SILICONE

	Tensile Strength psi	Hardness Shore A	Volume Resistivity and Thickness-in
Control	498	53 55	8.5×10^{12}
	480	53 55	
	460	54 54	0.093 0.097
	520	53.5	0.096 0.090
	611	56 av. 54	0.092 av. 0.094
After ETO - Freon 12 exposure	av. 493		
		54 59	1.3×10^{13}
		55 57	
		57	0.090 0.090
		57 av. 57	0.089 av. 0.090
After ETO - Freon 12 exposure and dry heat cycling	490	55 51	9.3×10^{12}
	510	54 51	
	502	53	0.089 0.091
	av. 500	53 av. 53	0.090 av. 0.090

ELASTOMERS

PR-1930-2, PRODUCTS RESEARCH, SILICONE

	Hardness Shore A	Volume Resistivity and Thickness-in.
Control	75 72	1.4×10^{13}
	75 72	
	71	0.080 0.081
	68 av. 72	0.087 0.086 0.083 av. 0.083
After ETO - Freon 12 exposure	72 72	9.6×10^{12}
	74	
	71 av. 72.5	0.082 0.082
	72	0.084 0.083
After ETO - Freon 12 exposure and dry heat cycling	76 76	1.0×10^{13}
	75 67	
	70	0.083 0.080
	71 av. 74	0.086 0.086 0.084 av. 0.083

2-218-S417-7, PARKER SEAL CO., SILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A
Control	865	600	73
	875	640	72
	890	610	70 av. 72
	970 av. 880	640	
After ETO - Freon 12 exposure	795	162	
	934	214	
	890	250	
	956 788 av. 870	258 206 av. 232	
After ETO - Freon 12 exposure and dry heat cycling	865	201	63
	845	204	63
	830	184	67 av. 64
	820 av. 840	131 av. 197	

RTV 560, GENERAL ELECTRIC, SILICONE

	Tensile Strength psi	Hardness Shore A	Volume Resistivity and Thickness-in.
Control	690	55 54	4.5×10^{13}
	600	54 53	
	680	54 54	0.067 0.069
	av. 653	53 av. 54	0.068 0.070 av. 0.067
After ETO - Freon 12 exposure		59 58	5.1×10^{13}
		59	
		58 av. 59	0.069 0.070
		60	0.069 0.068 av. 0.069
After ETO - Freon 12 exposure and dry heat cycling	710	62 62	2.4×10^{13}
	650	60 61	
	680	60	0.068 0.068
	av. 680	61 av. 61	0.074 0.072 0.065 av. 0.069

ELASTOMERS

RTV 615, GENERAL ELECTRIC, SILICONE

	Tensile Strength psi	Hardness Shore A	Volume Resistivity and Thickness-in.
Control	820	57 58	7.0×10^{13}
	850	58	
	960	58 av. 58	0.075
	880 av. 877	56	Hartshorn sample holder
After ETO - Freon 12 exposure		55 52	6.6×10^{13}
		54.5 55	
		51	
		48 av. 53	0.076
After ETO - Freon 12 exposure and dry heat cycling	260	66 63	1.4×10^{15}
	70	68 66	
	832	69 65	
	847	68	
	852	63 av. 64	0.075
	av. 844		

VITON B60, DU PONT, FLUOROCARBON

	Tensile Strength psi x 10^3	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in.
Control	3.42	350	74 74	6.5×10^{13}
	3.86	310	73 74	
	3.57	320	71	0.067 0.068
	3.56	av. 327	73 av. 74	0.069 0.064
	3.62			av. 0.067 0.067
	av. 3.52			
After ETO - Freon 12 exposure	2.77		74 74	4.9×10^{13}
	3.11		75 75	0.071 0.070
	2.94		74	0.071 0.069
	2.63		73 av. 74	0.067 av. 0.070
After ETO - Freon 12 exposure and dry heat cycling	3.15	240	76 77	7.1×10^{14}
	3.04	250	79 77	0.064 0.060
	2.83	240	76	0.062 0.0650
	av. 3.00	240	76 av. 77	av. 0.064 0.0690
		av. 242		

VITON B90, DU PONT, FLUOROCARBON

	Tensile Strength psi x 10^3	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in.
Control	2.34	125	93 93	2.8×10^{15}
	2.26	120	94 93	0.063 0.067
	2.14	115	93	0.062 0.068
	2.36	130	93 av. 93	0.064 av. 0.065
	2.50			
	av. 2.32	av. 123		
After ETO - Freon 12 exposure	2.01		93 91	6.6×10^{14}
	2.06		91 92	0.071 0.0660
	1.86		91	0.066 0.0698
	2.02 av. 1.96		92 av. 92	av. 0.068 0.0682
After ETO - Freon 12 exposure and dry heat cycling	2.62	100	94 93	6.9×10^{14}
	2.30	100	94 93	0.068 0.063
	2.56	115	95	0.064 0.064
	av. 2.49	av. 105	91 av. 93	av. 0.065 0.064

ENCAPSULANTS

RTV 11, GENERAL ELECTRIC, SILICONE

	Tensile Strength psi	Hardness Shore A	Volume Resistivity and Thickness-in
Control	410 460 400 av. 423	56 57 57 57 57 57 av. 57	3.0×10^{13} ----- 0.067 0.063 0.072 0.064 0.070 0.067 av.
After ETO - Freon 12 exposure		59 58 58 59 58 58 av. 58	2.0×10^{13} ----- 0.064 0.067 0.068 0.066 av. 0.066
After ETO - Freon 12 exposure and dry heat cycling	380 300 330 av. 337	59 59 59 58 59 59 av. 59	5.5×10^{15} 0.061 0.069 0.064 0.067 0.072 0.066 av.

RTV 60, GENERAL ELECTRIC, SILICONE

	Tensile Strength psi	Hardness Shore A	Volume Resistivity and Thickness-in
Control	340 390 320 av. 350	73 71 72 71 69 71 av. 70	6.3×10^{13} ----- 0.090 0.088 0.087 0.090 0.091 0.089 av.
After ETO - Freon 12 exposure		72 68 72 70 70 71.5 av. 69	5.1×10^{13} ----- 0.082 0.093 0.090 0.098 0.086 0.090 av.
After ETO - Freon 12 exposure and dry heat cycling	280 315 325 av. 307	72 73 74 72 71 73, 73 71 73 av.	1.5×10^{14} ----- 0.088 0.097 0.077 0.088 0.083 0.087 av.

ENCAPSULANTS

RTV 881, DOW CORNING, SILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity
Control	483 430 476 501 av. 472	190 190 185 190 av. 195	50 52.5 52 52 53 52 av.	6.8×10^{13} ----- 0.079 Hartshorn sample holder
After ETO - Freon 12 exposure	433 423 471 av. 442	155 160 170	56 58 57 57 55 56 av. 56	6.10×10^{13} ----- 0.081
After ETO - Freon 12 exposure and dry heat cycling	420 440 436 av. 432		62 61 62 60 58 62 60 61 av. 59	8.7×10^{13} ----- 0.078

STYCAST 1095, EMERSON AND CUMING, EPOXY

	Tensile Strength psi	Elastic Modulus $\times 10^5$	Hardness Shore D	Volume Resistivity
Control	780 810 1060 av. 883	4.84 5.20 4.85 4.99 av. 5.10	75 79 79 78 78 79 79 79 75 78 av.	9.3×10^{14} ----- 0.070 Hartshorn sample holder
After ETO - Freon 12 exposure	920 840 1220 av. 993	4.30 4.30 4.65 4.41 av. 3.45	77 88 79 85 78 86 79 79 av. 86	8.1×10^{14} ----- 0.071
After ETO - Freon 12 exposure and dry heat cycling	1020 1025 940 av. 1000		75 84 76 85 75 75.5 av. 86	1.3×10^{15} ----- 0.070

ENCAPSULANTS

STYCAST 2651/11, EMERSON AND CUMING, EPOXY

	Shear Strength psi x 10 ³	Hardness Shore D	Volume Resistivity and Thickness-in
Control	2.27 2.22 2.36 av. 2.29	84 85 85 86 87 87 90 86 av. 85	1.6 x 10 ¹⁵ ----- 0.75 Hartshorn sample holder
After ETO - Freon 12 exposure	2.42 2.12 2.62 av. 2.39	85 83 85 85 85.5 86 85 87.5 av.	9.2 x 10 ¹⁴ ----- 0.073
After ETO - Freon 12 exposure and dry heat cycling	2.79 2.70 2.76 av. 2.75	83 85 84 85 85 85 av. 88	2.3 x 10 ¹⁵ ----- 0.074 0.071 0.072 av.

SYLGARD 182, DOW CORNING, SILICONE

	Tensile Strength psi	Ultimate Elongation %	Hardness Shore A	Volume Resistivity and Thickness-in
Control	954 896 838 av. 896	175 150 150 av. 158	69 63 64 65 65 63 63 64 av. 64	6.0 x 10 ¹³ ----- 0.070 0.072 0.070 0.071 0.071 0.071 av.
After ETO - Freon 12 exposure	969 860 810 801 av. 860	250 160 155 150 av. 153	66 64 65 65 65 64 68 65 av. 64	4.0 x 10 ¹³ ----- 0.071 0.071 0.070 0.072 0.069 0.070 av.
After ETO - Freon 12 exposure and dry heat cycling	410 465 av. 431		69 70 69 66 68 69 av. 70	6.6 x 10 ¹³ ----- 0.070 0.070 0.067 0.071 0.069 0.069 av.

FILMS

H-FILM, DUPONT, POLYIMIDE

	Tensile Strength psi x 10 ³	Ultimate Elongation %	Volume Resistivity and Thickness-in
Control	26.0	65	1.9×10^{15}
	28.0	60	0.002 0.002
	26.0	55	0.002 0.002
	26.0	av. 60	0.003 0.002 av.
	24.0		
	av. 26.4		
After ETO - Freon 12 exposure	13.0		3.0×10^{17}
	12.5		0.002 0.003
	13.4		0.002 0.002
	av. 12.97		0.002 0.002 av.
After ETO - Freon 12 exposure and dry heat cycling	23.1	85	3.2×10^{17}
	23.8	90	0.002 0.002
	24.2	85	0.002 0.003
	av. 21.5	av. 85	0.002 0.002 av.

MYLAR 5 MIL- TYPE D, DUPONT, POLYESTER

	Tensile Strength psi x 10 ³	Ultimate Elongation %	Volume Resistivity and Thickness-in
Control	22.2	60	9.3×10^{16}
	22.1	60	0.006 0.006
	21.8	55	0.006 0.005
	24.3	70	0.006 0.006 av.
	23.1	65	
	av. 22.7	av. 62	
After ETO - Freon 12 exposure	21.2	70	9.3×10^{16}
	22.7	74	0.006 0.006
	20.8	72	0.005 0.007
	21.8	73	0.006 0.006 av.
	19.6	73	
After ETO - Freon 12 exposure and dry heat cycling	22.9	75.0	1.1×10^{17}
	21.4	70.0	0.006 0.006
	22.2	80.0	0.005 0.006 av.
	av. 22.17	av. 75.0	0.006

MYLAR 65 HS, DUPONT, POLYESTER

	Tensile Strength psi x 10 ³	Volume Resistivity
Control	7.42	7.4×10^{16}
	7.42	0.0012 0.0013
	7.23	0.0012 0.0013
	av. 7.35	0.0013 0.0013 av.
After ETO - Freon 12 exposure	11.84	8.3×10^{16}
	13.33	0.0013 0.0013
	12.33	0.0012 0.0013 av.
	13.33	0.0013
	13.03	
	av. 12.77	
After ETO - Freon 12 exposure and dry heat cycling	13.33	1.1×10^{17}
	15.71	0.0013 0.0013 av.
	12.11	0.0013
	12.11	0.0013
	15.15	
	12.63	
	av. 13.50	

FILMS

MYLAR 3 MIL, DUPONT, POLYESTER

	Tensile Strength psi x 10 ³	Ultimate Elongation %	Volume Resistivity and Thickness-in
Control	22.7 24.3 21.9 23.5 20.9 av. 22.6	80 85 80 80 av. 80	1.4×10^{16} 0.003 0.003 0.003 0.003 av. 0.003
After ETO - Freon 12 exposure	22.3 23.3 19.3 19.8 21.0 21.7 av. 21.2		1.1×10^{17} 0.003 0.003 0.003 0.003 av. 0.003
After ETO - Freon 12 exposure and dry heat cycling	15.49 16.63 15.92 av. 16.01	125 125 130 av. 125	1.7×10^{17} 0.003 0.003 0.003 0.003 0.004 0.003 av.

TEDLAR 200 TYPE 30B WHITE, DUPONT, POLYVINYL FLUORIDE

	Tensile Strength psi x 10 ³	Ultimate Elongation %	Volume Resistivity and Thickness-in
Control	14.0 14.0 14.0 14.0 14.0 av. 14.0	110 125 125 115 av. 119	2.8×10^{15} 0.002 0.002 0.002 0.002 av. 0.002
After ETO - Freon 12 exposure	10.16 9.68 10.88 av. 10.5	130 130 130 av. 130	1.9×10^{15} 0.002 0.002 0.002 0.002 av. 0.002
After ETO - Freon 12 exposure and dry heat cycling	11.0 12.0 11.8 12.0 9.9 av. 11.35	105 105 105 115 110 av. 108	2.7×10^{16} 0.002 0.002 0.002 0.002 av. 0.002

LUBRICANTS

DOW 200-350cs, DOW CORNING, SILICONE

	Viscosity Centistokes	Dielectric Breakdown Kv.
Control	325.9 cs 325.8 cs	18.0 17.0 16.0 av. 17.0
After ETO - Freon 12 exposure	354.8 354.9	18.4 18.0 16.0 17.3 av. 17.4
After ETO - Freon 12 exposure and dry heat cycling	403.4 403.3	17.0 14.5 15.8 av. 15.8

DC-5 GREASE, DOW CORNING, SILICONE

	Dielectric Breakdown Kv.
Control	17.6 16.9 11.8 13.3 14.4 av. 14.4
After ETO - Freon 12 exposure	11.6 12.8 av. 12.2
After ETO - Freon 12 exposure and dry heat cycling	11.6 10.8 11.2 av. 11.2

GE-F-50, GENERAL ELECTRIC, SILICONE

	Viscosity Centistokes	Dielectric Breakdown Kv.
Control	78.9 78.8 av. 78.8	21.0 16.5 17.0 av. 18.0
After ETO - Freon 12 exposure	62.6 63.0 63.3 av. 63.0	15.6 21.0 18.0 22.0 av. 19.1
After ETO - Freon 12 exposure and dry heat cycling	76.9 77.5 77.6 av. 77.3	15.2 12.4 18.4 14.4 av. 15.1

REINFORCED AND UNREINFORCED PLASTIC LAMINATES

DIAL FS-4 MESA PLASTICS, DIALLYL PHTHALATE

	Tensile and Flexural Strength T psi x 10 ³ F		Elastic Modulus x 10 ⁶	Hardness Shore D	Volume Resistivity and Thickness-in
Control	5.65	12.1	1.22	91 91	4.9 x 10 ¹³
	6.36	13.7	1.24	90 91	-----
	5.95		1.26	91 91	0.116 0.113
				90 91 av.	0.120 0.120
	av. 6.02	av. 12.9	1.24 av.		0.114 0.116 av.
After ETO - Freon 12 exposure	5.09	10.4	1.15	89 90	2.7 x 10 ¹³
	5.00	14.4	1.18	90 86	-----
				91 90	0.123 0.123
				86 89 av.	0.123 0.127
	av. 5.06	av. 12.4	1.16 av.		0.123 0.124 av.
After ETO - Freon 12 exposure and dry heat cycling	5.10	9.8		90 91	3.5 x 10 ¹³
	5.71	11.7		91 91	-----
	4.79	10.9		90 90.5 av.	0.119 0.116
	4.98			90	0.115 0.121
	av. 4.88	av. 10.8			0.118 0.118 av.

DIAL FS-10, MESA PLASTIC, DIALLYL PHTHALATE

	Tensile and Flexural Strength T psi x 10 ³ F		Elastic Modulus x 10 ⁶	Hardness Shore D	Volume Resistivity and Thickness-in
Control	8.92	14.9	1.62	91 90	1.4 x 10 ¹⁵
	9.12	14.05	1.63	90 91	-----
	8.86	14.5	1.61	91 91	0.130 0.128
	9.14			91 91 av.	0.130 0.128
	av. 9.00	av. 14.48	1.62 av.		0.133 0.129 av.
After ETO - Freon 12 exposure	8.80	13.1	1.50	94 91	3.56 x 10 ¹⁴
	9.78	17.3	1.61	90 92	-----
	9.37	15.0	1.54	90 90	0.125 0.128
				91 91 av.	0.12 0.130
	av. 9.31	av. 15.2	1.55 av.		0.129 0.128 av.
After ETO - Freon 12 exposure and dry heat cycling	7.8	13.6	0.6	91 91	2.0 x 10 ¹⁵
	7.9	12.09	0.6	90 90	-----
	7.0	12.55	0.6	90 92	0.130 0.131
				92 91 av.	0.130 0.132
	av. 7.5	av. 12.8	0.6 av.		0.130 0.130 av.

REINFORCED AND UNREINFORCED PLASTIC LAMINATES

EG 758T LAMINATE MIL-F-13949 FL-G.E. 062C, MICA CORP., EPOXY

	Tensile and Flexural Strength T psi x 10 ³ F		Elastic Modulus x 10 ⁶	Hardness Shore D	
Control	36.0 35.0 36.8 av. 62.8	62.2 62.9 63.3 av. 62.8	3.01 3.04 3.00 2.91 3.00 av.	83.5 84 84 83 83	83 84 83 83 av.
After ETO - Freon 12 exposure	45.2 44.9 45.3 av. 43.0	61.5 61.9 61.5 av. 61.5	2.91 2.89 2.83 2.81 2.86 av.	84 82 81 83	83 84 83 av.
After ETO - Freon 12 exposure and dry heat cycling	37.4 38.8 39.0 38.6 av. 38.4	54.7 55.5 54.7	1.9 1.9 2.1 1.9 2.0 av.	84 86 87 81 83	85 83 82 85 av.

FIBERGLAS 91LD, AMER. REINF. PLASTICS, PHENOLIC

	Tensile and Flexural Strength T psi x 10 ³ F		Elastic Modulus x 10 ⁶	Ultimate Elonga- tion %	Hardness Shore D	Volume Resistivity and Thickness-in	
Control	32.0 30.1 32.0 32.0 av. 32.0	51.7 59.1 44.6 av. 55.4	2.9 3.0 2.6 2.8 av.	1.6 1.5 1.6 1.6 1.6 av.	90 91 90 90 90 av.	4.5 x 10 ¹³	0.071 0.070 0.072 0.071 av.
After ETO - Freon 12 exposure	34.5 35.4 35.7 36.8 av. 35.6	60.0 63.2 63.8 av. 61.0	2.79 3.09 2.78 2.93 2.9 av.		91 92 91 90 90 av.		0.066 0.069 0.065 0.067 av.
After ETO - Freon 12 exposure and dry heat cycling	29.4 29.1 29.4 30.6 av. 29.6	51.4 53.5 57.5 62.2 av. 54.1	2.77 2.96 2.72 2.72 2.79 av.		92 90 93 92 92 av.	7.6 x 10 ¹³	0.058 0.063 0.060 0.061 av.

REINFORCED AND UNREINFORCED PLASTIC LAMINATES

LAMINATE EG 752, MICA CORP.

	Tensile and Flexural Strength psi		Elastic Modulus $\times 10^6$	Hardness Shore D		Volume Resistivity and Thickness-in
	T	F				
Control	41.7	52.0	5.59	92	91	2.4×10^{13} ----- 0.021 0.020 0.020 0.020 av. 0.020
	45.5	59.1	5.35	91	92	
	43.3	44.8	5.08	92	92 av.	
	44.6	54.5	5.08	93		
	43.2	47.6	6.10			
	av. 43.6	av. 51.6				
After ETO - Freon 12 exposure	40.2			92	92	1.2×10^{14} ----- 0.019 0.020 0.020 0.020 0.021 0.020 av.
	37.1			92	92	
	41.3			91	92 av.	
	av. 39.5			91		
After ETO - Freon 12 exposure and dry heat cycling	39.9			93	91	9.2×10^{12} ----- 0.020 0.019 0.019 0.020 0.019 0.020 av.
	38.3			93	92	
	34.7			92	92 av.	
	av. 37.6			92		

H-5834 MIL-R-9299, TYPE II, CL 1, WESTINGHOUSE, PHENOLIC

	Tensile and Flexural Strength psi		Elastic Modulus $\times 10^6$	Hardness Shore D		Volume Resistivity and Thickness-in
	T	F				
Control	51.5	47.7	3.0	93	93	4.0×10^{14} ----- 0.068 0.067 0.068 0.067 0.067 0.067 av.
	53.3	68.0	3.44	92	94	
	52.2	69.0	3.35	91	93 av.	
	52.5	71.7	3.35	93		
	51.0	65.2	3.29			
	av. 52.1	av. 64.3	3.29 av.			
After ETO - Freon 12 exposure	48.3	67.7	3.19	93	93	3.0×10^{14} ----- 0.067 0.067 0.068 0.067 0.067 0.067 av.
	49.8	67.2	3.13	91	93	
	52.0	66.3	3.25	90	92.5 av.	
	48.6	63.6	3.28	93		
	53.1	67.9				
	av. 52.1	av. 64.3	3.21 av.			
After ETO - Freon 12 exposure and dry heat cycling	49.4	69.2	3.39	93	93	1.7×10^{14} ----- 0.067 0.068 0.067 0.070 0.068 0.068 av.
	51.3	67.8	3.13	94	92	
	50.4	75.2	3.12	92	93 av.	
	53.3	69.6	3.15	93		
	52.1	70.5	3.02			
	av. 51.3	av. 68.5	3.16 av.			

REINFORCED AND UNREINFORCED PLASTIC LAMINATES

LAMINATE NS MIL-P-15047, PLASTIC CENTER INC., PHENOLIC

	Tensile and Flexural Strength psi x 10 ³ T F		Elastic Modulus x 10 ⁶	Hardness Shore D		Volume Resistivity and Thickness-in
Control	6.38	7.57	0.277	85	85	1.9 x 10 ¹⁴
	6.21	9.08	0.264	85	86	
	6.13	8.35	0.250	85	86	0.063 0.064
	6.25	8.67	0.251	86	86.0 av.	0.064 0.064
	6.97	7.88	0.255	85		0.062 0.063 av.
	av. 6.39	av. 8.70	0.260 av.			
After ETO - Freon 12 exposure	6.82	7.58	0.276	85	85	7.18 x 10 ¹³
	6.54	8.95	0.293	86	86	
	7.04	8.10	0.285	85	85.5 av.	0.063 0.063
	7.07	7.37		86		0.064 0.063
	6.62	7.45				0.063 0.063 av.
	av. 6.81	av. 7.89	0.285 av.			
After ETO - Freon 12 exposure and dry heat cycling	6.73	11.4	0.412	86	88	2.86 x 10 ¹⁴
	6.58	11.7	0.402	87	87	
	6.53	12.6	0.417	88	88 av.	0.064 0.064
	6.72	11.7	0.390	89		0.064 0.062
	6.98	11.35	0.405			0.062 0.063 av.
	av. 6.70	av. 11.8	0.405 av.			

LAMINATE 500J (MIL-P-13949B, FL-GE-C2/2), BUDD CO., EPOXY

	Tensile and Flexural Strength psi x 10 ³ T F		Elastic Modulus x 10 ⁶	Hardness Shore D		Volume Resistivity and Thickness-in
Control	47.4	71.0	3.59	90	91	9.5 x 10 ¹⁴
	48.2	68.0	3.44	86	91	
	48.3	69.6	3.43	90	89 av.	0.064 0.064
	47.9	95.5	3.49	90		0.065 0.064 av.
	45.6	93.0	3.48			0.063
	av. 47.5	av. 79.4	3.48 av.			
After ETO - Freon 12 exposure	47.6	74.3	3.56	91	91	2.6 x 10 ¹⁵
	47.6	93.2	3.74	91	91	
	51.3	87.3	3.76	91	90 av.	0.064 0.065
	49.8	91.0	3.54	91		0.065 0.065
	51.0	94.0				0.064 0.065 av.
	av. 49.4	av. 88.0	3.70 av.			
After ETO - Freon 12 exposure and dry heat cycling	45.9	93.3	3.29	91	90.5	1.4 x 10 ¹⁵
	47.2	90.4	3.23	90	91	
	50.0	89.0	3.08	91	91 av.	0.064 0.064
	47.4	90.7	3.73			0.064 0.063
	46.4	92.0	3.39			0.064 0.064 av.
	av. 47.4	av. 91.0	3.54 av.			

REINFORCED AND UNREINFORCED PLASTIC LAMINATES

MICARTA H-2497 MIL-P-18177 TYPE GEB, EPOXY - GLASS FIBER

	Tensile and Flexural Strength psi x 10 ³ T F		Elastic Modulus x 10 ⁶	Ultimate Elonga- tion %	Hardness Shore D		Volume Resistivity and Thickness-in
Control	45.3	73.8	2.86		84	84	4.9 x 10 ¹⁵
	45.0	73.6	2.86		81	85	
	45.7		2.82		82	84	0.064 0.064
	44.3		2.82		83	85	0.066 0.064
	47.0				81	84.5 av.	0.064 0.065 av.
	av. 45.3	av. 73.7	2.84 av.		85		
After ETO - Freon 12 exposure	44.2	72.0	2.79				6.9 x 10 ¹⁵
	45.0	72.0	2.77				
	45.0	72.0	2.75				0.069 0.069
	av. 44.7	av. 72.0	2.77 av.				0.069 0.069
							0.068 0.069 av.
After ETO - Freon 12 exposure and dry heat cycling	45.0	58.2		2.0	84	84	7.3 x 10 ¹⁵
	42.0	63.0		1.9	85	84	
	44.3	65.3		2.0	84	85	0.062 0.061
	av. 43.8	av. 61.5		2.0 av.	84	84 av.	0.062 0.062 av.
							0.062

MICARTA LE-221 (MIL-D-15035, TYPE FBE), WESTINGHOUSE

	Tensile and Flexural Strength psi x 10 ³ T F		Elastic Modulus x 10 ⁶	Hardness Shore D		Volume Resistivity and Thickness-in
Control	8.56	15.8	0.740	90	90	1.0 x 10 ¹⁴
	8.40	15.8	0.755	90	90	
	8.75	17.7	0.768	90	90 av.	0.062 0.060
	8.78	17.15	0.768	90		0.061 0.060
	8.53	15.85	0.740			0.060 0.062 av.
	av. 8.6	av. 16.5	0.755 av.			
After ETO - Freon 12 exposure	9.02	15.9		90	90	1.1 x 10 ¹⁴
	9.00	17.1		90	90	
	8.92	14.6		91	90 av.	0.063 0.061
	8.92	16.3		90		0.063 0.062
	8.77	15.5				0.062 0.062 av.
	av. 8.92	av. 16.0				
After ETO - Freon 12 exposure and dry heat cycling	9.15	13.9	0.764	91	91	4.3 x 10 ¹⁵
	9.27	14.8	0.80	91	91	
	9.32	13.8	0.805	90	91 av.	0.062 0.061
	9.50	16.5	0.755	91		0.062 0.062
		16.1				0.062 0.062 av.
	av. 9.31	av. 15.0	0.781 av.			

REINFORCED AND UNREINFORCED PLASTIC LAMINATES

MICARTA 238 MIL-P-15035 TYPE FBI, WESTINGHOUSE, PHENOLIC

	Tensile and Flexural Strength psi x 10 ³ T F		Elastic Modulus x 10 ⁶	Hardness Shore D	Volume Resistivity and Thickness-in
Control	10.2	18.7	0.903	90 90	3.5 x 10 ¹²
	11.2	18.1	0.928	90 90	
	11.15	17.9	0.862	90 90 av.	0.065 0.065
	10.45	17.2	0.873		0.065 0.065
	11.00	18.0	0.958		0.065 0.065 av.
	av. 10.8	av. 18.0	0.905 av.		
After ETO - Freon 12 exposure	10.4	16.5	0.945	90 90	1.24 x 10 ¹²
	11.6	17.0	0.840	90 90	
	11.25	17.9	0.840	89 90 av.	0.065 0.063
	10.75	16.7	0.875 av.	90	0.065 0.063
	11.0				0.063 0.064 av.
	av. 11.00	av. 17.0			
After ETO - Freon 12 exposure and dry heat cycling	11.3	19.2	0.923	91 91	2.4 x 10 ¹²
	12.0	16.3	1.01	91 91	
	11.9	17.2	0.970	92 91 av.	0.065 0.060
	10.7	18.2	0.860	91	0.065 0.064
	11.6	18.6	0.905		0.063 0.064 av.
	av. 11.5	av. 17.9	0.933 av.		

MICARTA GX (H17480) MIL-P-18177 GEE, WESTINGHOUSE, EPOXY

	Tensile and Flexural Strength psi x 10 ³ T F		Elastic Modulus x 10 ⁶	Hardness Shore D	Volume Resistivity and Thickness-in
Control	60.3	96.0	2.96	91 88	6.1 x 10 ¹³
	55.8	98.5	2.96	91 89	
	56.1	98.5	3.04	90 90 av.	0.060 0.059
	55.7	91.6	2.95	89	0.059 0.061
	57.7	95.0	3.35		0.059 0.060 av.
	av. 55.1	av. 96.0	3.05 av.		
After ETO - Freon 12 exposure	59.3	92.8	2.65	91 88	9.45 x 10 ¹³
	60.5	88.0	2.62	90 89	
	54.5	91.0	2.59	90 90 av.	0.059 0.059
	60.3	93.5	2.63	90	0.061 0.060
	60.0	89.5	2.66		0.063 0.060 av.
	av. 58.9	av. 90.9	2.63 av.		
After ETO - Freon 12 exposure and dry heat cycling	58.8	101.5	2.51	91 89	3.0 x 10 ¹⁴
	61.4	93.8	2.65	91 89	
	43.8	89.4	2.65	87 89 av.	0.063 0.063
	63.0	91.5	2.71	88	0.060 0.059
	62.1	96.7	2.68		0.061 0.061 av.
	av. 58.0	av. 94.6	2.64 av.		

REINFORCED AND UNREINFORCED PLASTIC LAMINATES

MICARTA 8457 G-D, WESTINGHOUSE

	Tensile and Flexural Strength psi x 10 ³ T F		Elastic Modulus x 10 ⁶	Hardness Shore D		Volume Resistivity and Thickness-in
Control	46.1 44.8 46.8 43.8 43.6 av. 45.0	62.0 62.7 63.0 62.8 63.6 av. 62.8	2.99 3.15 3.01 2.99 1.94 2.81 av.	90 90 90 91	90 91 90 av.	1.5 x 10 ¹⁵ ----- 0.063 0.063 0.064 0.063 av. 0.063
After ETO - Freon 12 exposure	45.8 48.5 43.7 46.1 46.9 av. 45.8	63.3 66.0 66.0 67.7 63.5 av. 65.3	2.84 2.98 3.06 2.96 av.	90 92 90 90	90 91 91 av.	9.1 x 10 ¹³ ----- 0.062 0.063 0.062 0.063 0.062 0.063 av.
After ETO - Freon 12 exposure and dry heat cycling	45.6 47.0 46.2 44.4 46.5 av. 46.0	64.4 63.5 63.1 65.8 56.2 av. 60.2	2.98 2.98 2.91 2.88 2.99 2.94 av.	90 91 91 92	92 90 91 av.	7.2 x 10 ¹⁴ ----- 0.063 0.063 0.063 0.063 0.064 0.063 av.

XP-206 MINNESOTA MINING MFG., EPOXY

	Tensile and Flexural Strength psi x 10 ³ T F		Elastic Modulus x 10 ⁶	Hardness Shore D		Volume Resistivity and Thickness-in
Control	47.2 43.4 45.2 Av. 45.2	12.1 10.6 12.2 av. 12.1	2.27 2.27 2.26 2.27 av.	35 37 34 34	34 34 35 34 av.	4.7 x 10 ¹⁵ ----- 0.058 0.055 0.058 0.057 0.051 0.058 av.
After ETO - Freon 12 exposure	47.1 45.0 av. 46.0		2.37 2.32 2.35 av.			4.7 x 10 ¹⁵ ----- 0.060 0.058 0.058 0.058 0.058 0.059 av.
After ETO - Freon 12 exposure and dry heat cycling				36 35 35 34 34	34 34 34 34 av.	5.5 x 10 ¹⁵ ----- 0.059 0.052 0.057 0.053 0.052 0.055 av.